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*Minimizing Oil Loss in Miscella Refining*

**Stan C. Loft**

Loft Consulting Services, Inc.

San Diego, CA

# 46th Oilseed Conference

**Processing Efficiency:  
Meeting the Challenge**

**March 9–11, 1997**

Hotel Monteleone

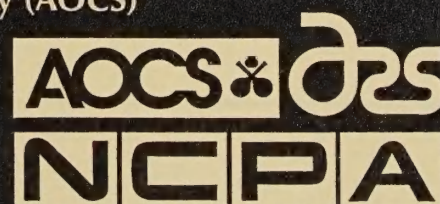
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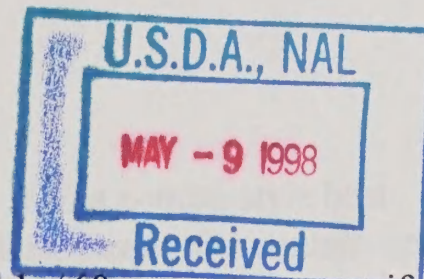






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*Specialists in Edible Oil Processing*



## **Why Miscella Refine?**

With the development of the Hermetic separator in the early '60s, one stage centrifugal separation was achieved because of the large difference in specific gravity's of the two phases. Water washing was eliminating therefore the need for waste water treatment.

Higher quality oil could be achieved through lower temperature processing. The dark color associated with Cottonseed Oil could be easily removed before a color 'set' from high temperature desolventizing. The oil processed is freshly extracted without the deleterious effect from long term storage. The Miscella Refinery is hermetic sealed with very little exposure to air or oxygen.

Higher Yields can be achieved because of the higher separation efficiencies and more efficient use of caustic and other reagents.

Simple method of soapstock disposal by adding it directly to the DT where residual hexane is extracted. The soapstock adds 'fat' value to meal which is normally added at the feed mill. The meal is also easier to pelletize without the usual dust problems

There is less energy required. The low temperature processing consumes one tenth of the steam required for conventional refining. The Horsepower is reduced by one third due to fewer motors and low viscous material. A 60% miscella at 100 F has a viscosity of 2.6 cp compared to crude Cottonseed Oil with 33 cp viscosity at the same temperature. Less operating staff is required since the process is considered an integral part of extraction.

## **Process Description (Fig. 1)**

Miscella from the first stage evaporator is stored temporarily in Work or Shift tanks where oil analysis are completed prior to processing. The Miscella is usually screened using duplex strainers that are lined with a fine wire cloth in order to remove as much meal as possible. Meal fines will eventually deposit on metal surfaces such as the centrifuge's disk stack which then requires frequent cleaning. A well designed Hydro cyclone would be more effective upstream of the first stage evaporator.

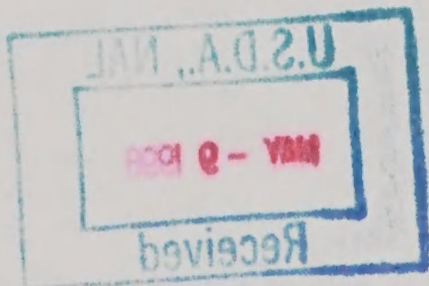
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MISCELL2.DOC





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The crude miscella is heated (or cooled) to 135 F to 140 F with a combo-style heat exchanger that will either cool or heat the product. Since the miscella can contain up to 0.2% meal fines, a straight tube exchanger is recommended with the miscella on the tube side and velocities exceeding 4 ft./sec.

The miscella is then pretreated with 50 to 500 ppm of Glacial Acetic acid or an inorganic acid injected and intensely dispersed. Very little reaction time is required at these temperatures. Pretreatment is not always required. However, provisions should be available for the darker oils

A Dilute solution of Sodium Hydroxide is injected and also intensely dispersed into the miscella. The reaction between the FFA and caustic occurs immediately producing soap miscelles. These micelles have a high affinity for each other and quickly agglomerate to form larger and dense clusters with very little oil occlusion.

The mixture passes through the Retention or Contactor mixers which are designed to continue the agglomeration process. It is in this section of the process where the dark colors such as Gossypol are adsorbed by the soap micelles. Phospholipids such as Phosphatidic acid are crystallized, as discussed earlier, and adsorbed on the soap miscelles. Early work by Crauer & Pennington<sup>1</sup> illustrated these formations with magnified photography. Photo #1 from their paper illustrates the crystalline form of phosphaditic acid. the crystals that appear are 1-2 microns in width and 8-12 microns long. The Phosphatidic content of the crude oil will influence the retention time required for their removal.

From the Retention Mixers, the mixture of miscella and soapstock is sent directly to the centrifuge. In the past, a trim heat exchanger was used to raise the mixture's temperature 5-10 F depending on the heat loss through the system. With a well insulated process, this trim heater is not required. In fact, the less turbulence to the fragile soap micelles formed in the retention section, the better the centrifugal separation.

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<sup>1</sup> 'Continuous Refining of Crude Cottonseed Miscella' presented by L.S. Crauer and H. Pennington; 1963 AOCS Convention

The Board of Directors of the Company has approved the payment of a dividend of \$1.00 per share of common stock for the quarter ended March 31, 1964. The dividend will be paid on or about April 15, 1964, to shareholders of record as of March 15, 1964.

The Board of Directors has also approved the payment of a dividend of \$0.50 per share of preferred stock for the quarter ended March 31, 1964. The dividend will be paid on or about April 15, 1964, to shareholders of record as of March 15, 1964.

The Board of Directors has further approved the payment of a dividend of \$0.25 per share of common stock for the quarter ended March 31, 1964. The dividend will be paid on or about April 15, 1964, to shareholders of record as of March 15, 1964.

The Board of Directors has also approved the payment of a dividend of \$0.125 per share of common stock for the quarter ended March 31, 1964. The dividend will be paid on or about April 15, 1964, to shareholders of record as of March 15, 1964.

The Board of Directors has further approved the payment of a dividend of \$0.0625 per share of common stock for the quarter ended March 31, 1964. The dividend will be paid on or about April 15, 1964, to shareholders of record as of March 15, 1964.

Very truly yours,



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The most essential part of Miscella Refining is the centrifuge where the soapstock is separated from the miscella. For more than 30 years, the hermetic separator from Alfa Laval has been the most successful. From the early VO-194s to the more current SMG-509, this separator, under the proper operation conditions, can produce a light phase discharge containing less than 50 ppm residual soap with less than 10% neutral oil in the soap phase. Compare these results to conventional refining where the residual soaps range between 100 - 500 ppm and the oil content of the soapstock exceeds 30%. These results are **before** Water washing.

The separator is the limiting factor in Miscella Refining capacity. The 'Hermetic Advantage' stems from the fact that the miscella is contained within the separator proper by the use of mechanical seals on both the inlet and outlets. The second 'Advantage' which this writer feels is most important is the ability to adjust the separation zone in the separator by adjusting the light phase back pressure. The open style centrifuge cannot offer this feature and must be cleaned frequently.

From the separator, the miscella can either be desolventized and stored as PBSY or bleached (in miscella) if the oil is going to be deodorized on site or by the end user. An example of a simple Miscella Bleach Plant is shown in Fig. 2.

The soapstock, containing 5 - 7% hexane, is pumped directly to the Desolventizer Toaster when it mixed and desolventized with the meal. The soapstock can also be acidulated and desolventized for fatty acid recovery should the market demand increase for this product.

### **What are the Controlling parameters for maximum Yields?**

Maintain a constant miscella concentration. The difference in specific gravity's between a 50% and 65% miscella concentration is 13%. This small differential will have a dramatic effect in where the separation occurs in the centrifuge. Repeatable miscella concentration can be accomplished by close temperature control of the 1st stage evaporator or by continuously adding back hexane to the miscella as it is pumped to the Shift tanks. The concentration can easily be controlled with an on-line densitometer.

# Left: Unpublished Manuscript

## General Introduction

The first section of the manuscript is a general introduction to the subject of the study. It discusses the importance of the research and the objectives of the study. The introduction is divided into two main parts: a general overview of the subject and a specific overview of the study. The general overview discusses the importance of the research and the objectives of the study. The specific overview discusses the objectives of the study and the methods used to achieve them.

The second section of the manuscript is a detailed description of the methods used in the study. It discusses the data collection methods, the data analysis methods, and the results of the study. The methods section is divided into three main parts: a description of the data collection methods, a description of the data analysis methods, and a description of the results of the study.

The third section of the manuscript is a discussion of the results of the study. It discusses the findings of the study and the implications of the findings. The discussion is divided into two main parts: a discussion of the findings and a discussion of the implications of the findings.

The fourth section of the manuscript is a conclusion of the study. It summarizes the findings of the study and the implications of the findings. The conclusion is divided into two main parts: a summary of the findings and a summary of the implications of the findings.

## References

The references section of the manuscript lists the sources of information used in the study. It includes a list of books, articles, and other sources of information. The references are listed in alphabetical order of the author's name.



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It is best to isolate crude miscella in agitated eight hour Shift tanks to allow time for oil analysis. This will enable the operator to establish the operating parameters such as pretreatment and caustic treat **before** the Shift tank is refined. Until on-line analytical instrumentation is available, each tank must be analyzed for theoretical loss (or cup loss), FFA, color and hopefully, Phosphatidic content.

As was stated earlier, acid pretreatment is not always necessary. However, it serves several purposes;

- With all processing parameters remaining constant, higher acid dosages will usually reduce color with dark or Green oils.
- Non-Hydratable Phospholipids (NHP) will precipitate to their respective Phosphatidic acids are removed with the soapstock and,
- The pretreatment will help compact the soap miscelles thus occluding neutral oil.

Caustic strength and excess can be two of the largest contributors to high losses. Typically, a low caustic concentration between 12% and 18% will yield better results. Higher strengths are used for difficult color removal. However, with a suitable pretreatment reagent, good color can be achieved using normal caustic strengths. Excess caustic should range between 0.2 and 0.3%. A low excess treat can be as detrimental as too much caustic.. Low excess will inhibit Gossypol removal and will not precipitate the Phosphatidic Acid that has been conditioned by pretreatment. Too much excess will lead to over-saponification and possible three-phasing in the separator.

Retention time is required to allow for excess caustic to react with the Phosphatides and Gossypol in order to deposit these complex precipitates on the soap miscelles. The time increment is directly proportional to the amount of NHP in the crude oil. Normally three to six minutes is sufficient; preferably in multiple retention mixers. Color reduction is normally the first indication of sufficient retention time. Added retention time serves no useful purpose and can actually be deleterious because of the oxidizing nature of the chemicals.

Separation is the key component an efficient Miscella Refinery. Keeping the separator clean and free of meal fines is paramount. If constant cleaning is affecting the operation





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downtime, perhaps a Clean-In-Place (CIP) system is warranted. An acid based detergent is first circulated through the centrifuge as a 'Forward' flush. The flow is then reversed 'Back' flushing the centrifuge. This step is most important as it **reverses** the flow through the disk stack dislodging imbedded meal fines that accumulate on the outer periphery of the disks. The detergent and solids are removed with a final hot water flush and the machine place back in service without dismantling.

### **Analytical Tools Available**

The high speed 10cc Gyrotester centrifuge is still one of the easiest methods for the operator to monitor separation efficiency. Light phase samples are analyzed for soap content down to 25 ppm. If however, if the sample is brilliantly clear with only a trace of solids in the test tube, there is a good chance of excessive neutral oil carryover with the soapstock. Samples can be drawn after each retention mixer to determine color removal and how the soapstock compacts in the bottom of the sample tube. Samples taken of the centrifuge feed mixture can determine caustic over-treatment as there will be a 'middle' phase formed which indicates free or un-reacted caustic. This condition will cause the separator to 'Three Phase' which will eventually restrict the flow of soapstock exiting the centrifuge.

We have developed a Yield Control and Monitoring system which measures the loss across the centrifuge instantaneously. 'Loss' systems have been around for decades and serve as a useful tool when optimizing refinery yields. This one goes a step further; we can factor out the hexane solvent to produce a 'Net' Yield on an oil basis.

There is also available a continuous In-Line Caustic dilution system which combined with the Yield System will not only allow the operator to change caustic strengths 'On-Line', but give the ability to control caustic excess by simply entering the FFA content of the Crude oil. The microprocessor computes the caustic treat based on the strength of caustic desired, the FFA and the excess amount entered by the operator. The prototype for this system is currently under test and evaluation in a large miscella refinery. We should have final evaluation by the end of this year.

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## **Summary**

Miscella Refining, because of its simple nature and efficiencies, has the potential to become the Edible Oil Process of decade. Already, there processors who refine Safflower in miscella. Plant tests were successful refining fresh Soybean miscella during the late sixties. Corn and Sunflower has also been tested in miscella. The missing ingredient is a larger centrifuge that will match the efficiencies of what we have today. This is the Challenge.







PHOTO #1





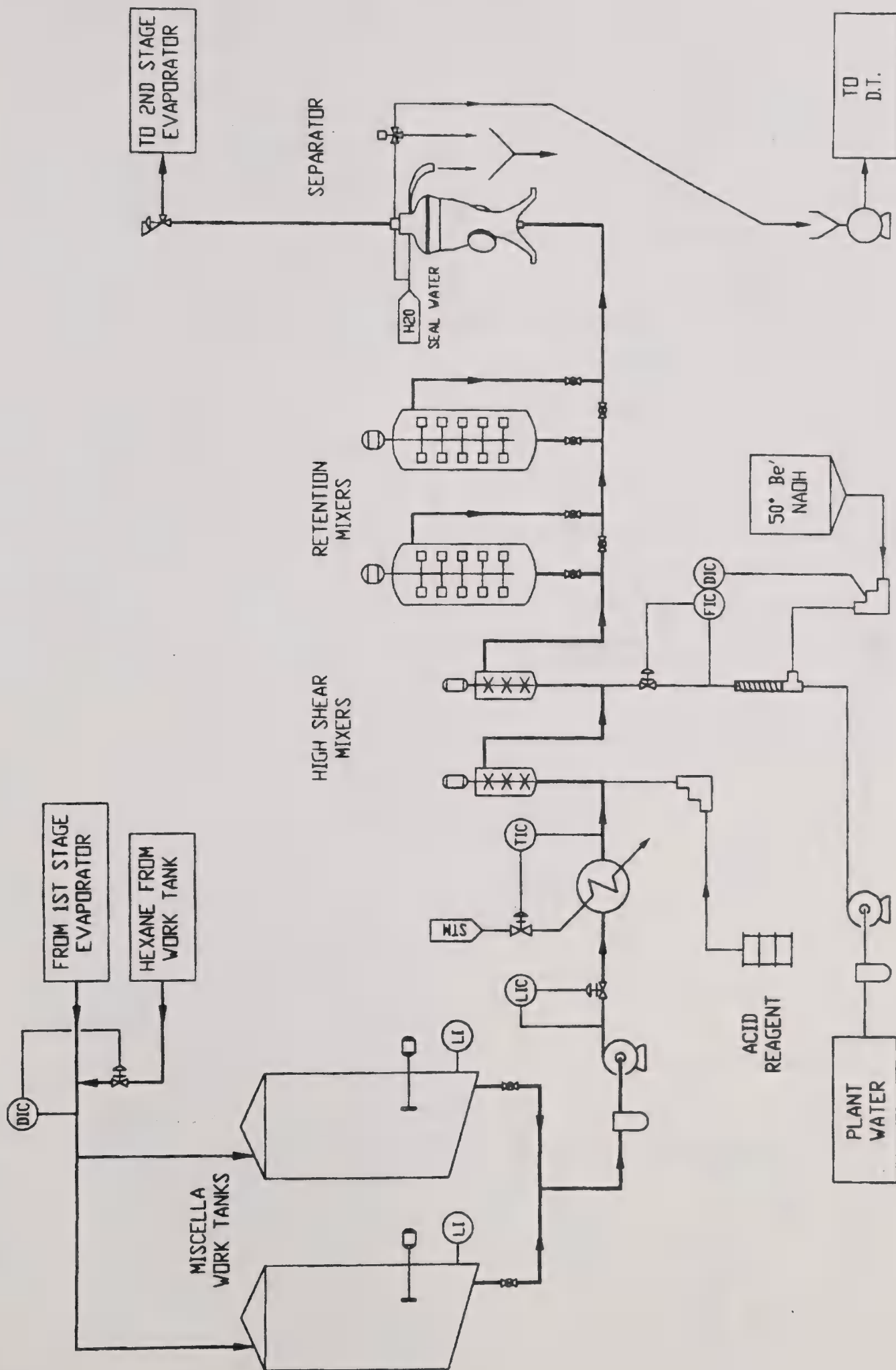


FIG. 1



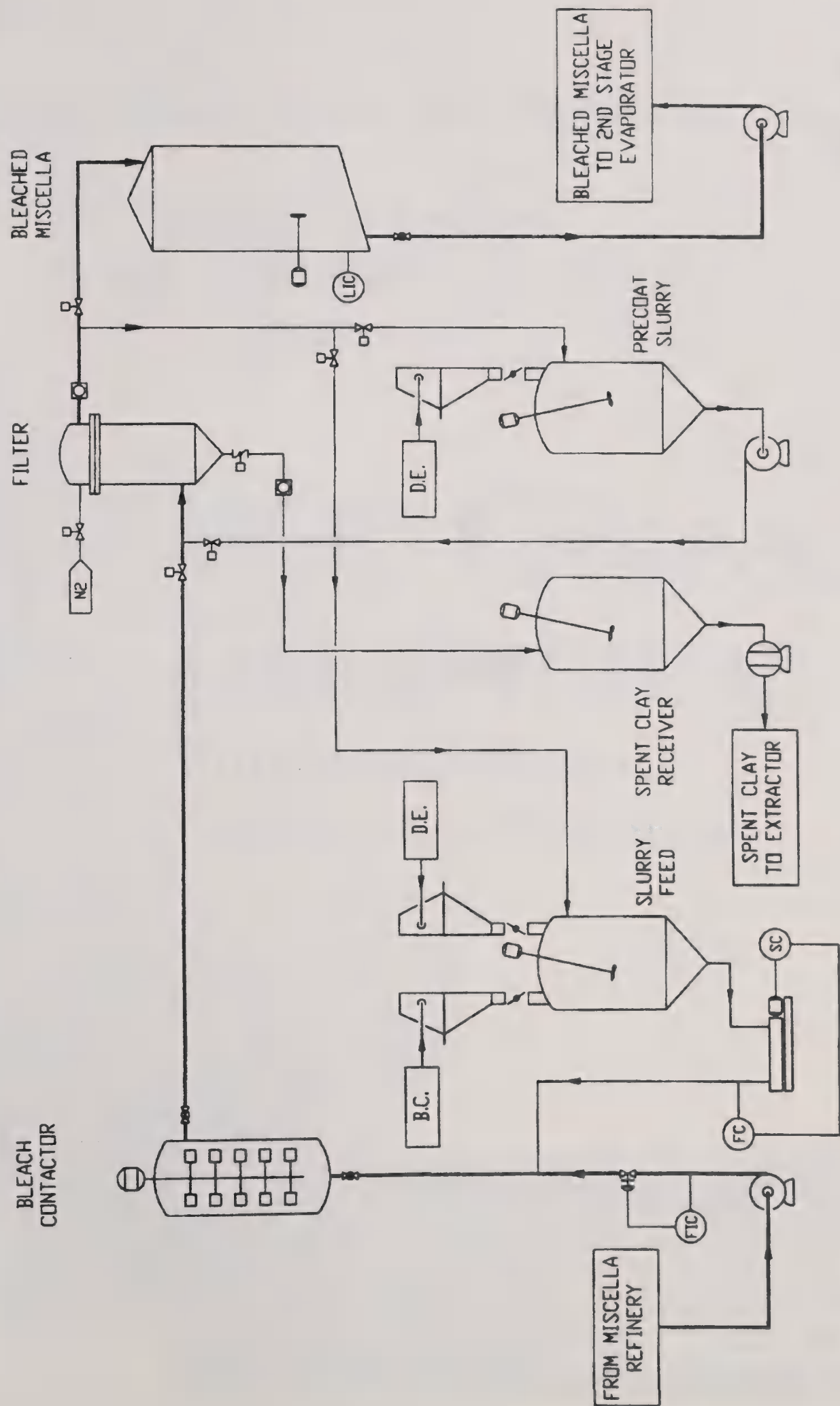


FIG. 2





*Minimizing Oil Loss in Miscella Refining*

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French Oil Mill Machinery Co.  
Piqua, OH

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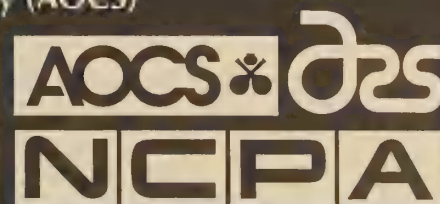
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# Minimizing solvent loss

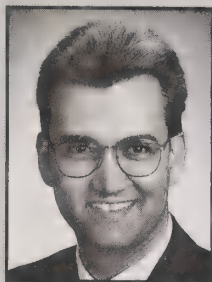
As the next century approaches, industry is fully engaged in quality, knowing that continuous improvement is an essential strategy for future prosperity. Quality also applies to our environment. We must continuously improve our processes to reduce the negative impact imparted on the environment.

For the oilseed processing industry, one of the negative impacts imparted on the environment is the unrecovered solvent from the vegetable oil extraction process. The solvent most commonly used in this process is hexane, which has been identified as a hazardous air pollutant by recent environmental regulations. The oilseed processing industry must strive to continuously improve solvent recovery to improve the quality of the environment we live in.

In the early years of solvent extraction of vegetable oil from oilseeds, it was considered "good" solvent recovery in a soybean solvent extraction plant to recover 99.72% of the solvent pumped into the extractor. This rate of recovery appeared both reasonable and economically feasible. In other terms, approximately 1.0 gallon of solvent was lost for every ton of soybeans processed.

In the 1970s, with the advent of larger plants and new technology for scrubbing solvent from the exiting process air, what was considered "good" solvent recovery in a soybean solvent extraction plant improved to 99.86% of the solvent pumped into the extractor. In other terms, the solvent loss was reduced to approximately 0.5 gallons of solvent per ton of soybeans processed.

This remained what was considered "good" solvent recovery until the 1980s and 1990s, when plants installed improved desolventizer toasters utilizing fully countercurrent stripping steam. Presently, most processors considered "good" solvent recovery in soybean solvent extraction plants to be 99.92% of the solvent pumped into the extractor, resulting in a loss of approximately 0.3 gallons of



*This article was written by Timothy G. Kemper, director of engineering for the Oilseed Division of French Oil Mill Machinery Co., 1035 W. Greene St., Piqua, Ohio 45356.*

solvent per ton of soybeans processed.

To maximize solvent recovery in a modern soybean solvent extraction plant, the following technology must be in place:

## A. Desolventizer Toaster

- fully countercurrent, evenly distributed live steam flow
- superheat in steam supply (350°F @ 20 psig)
- adequate discharge meal temperature (220°F or higher)
- adequate residence time (20 minutes or more)

## B. Mineral Oil System

- low entering vapor temperature (100°F or lower)
- sufficient scrubbing contact area (see maximum design vapor flow)
- sufficient stripping contact area (see maximum design vapor flow)
- sufficient mineral oil flow (see minimum design oil flow)
- low enough cool mineral oil temperature (95°F or lower)
- high enough hot mineral oil temperature (220°F or higher)
- fully countercurrent, evenly distributed vapor to oil flow

- superheat in steam supply (350°F @ 0 psig)
- good quality mineral oil

## C. Final Oil Stripper

- adequate oil concentration in (97% oil or higher)
- adequate oil temperature in (220°F or higher)
- fully countercurrent, evenly distributed live steam flow
- superheat in steam supply (350°F @ 0 psig)
- adequate stripping contact area (see maximum design oil flow)
- adequate vacuum level (24 inches Hg or higher at sea level)

## D. Waste Water Evaporator

- adequate water temperature (185°F or higher)
- adequate residence time (20 minutes)

With properly sized, latest technology equipment in place under consistent operation conditions, solvent loss assumptions can be made for a soybean solvent extraction plant (Table 1).

*(continued on page 900)*

**Table 1**  
**Solvent loss assumption for soybean solvent extraction facility**

Source of loss	Amount of loss
In meal from desolventizer toaster	400 ppm
In meal from dryer cooler	100 ppm
In air from mineral oil system	30% lower explosive limit (LEL) of hexane mixed with air
In oil from final oil stripper	200 ppm
In water from waste water evaporator	10 ppm

**Table 2**  
**Solvent loss analysis for soybean solvent extraction facility**

Source of loss	Amount of loss (gallons per ton)
In meal from dryer cooler	0.028
In air from meal dryer cooler	0.083
In air from mineral oil system	0.021
In oil from final oil stripper	0.013
In water from waste water evaporator	0.002
Total accountable solvent loss	0.147

Note: Soybean is the easiest material to desolventize, and as a result, solvent loss in other oilseeds will be greater in the areas of air from the meal dryer cooler, and in meal from the meal dryer cooler.

(continued from page 898)

Table 2 shows a solvent loss analysis when these solvent losses are applied to the mass balance of a soybean solvent extraction plant.

Assuming good solvent loss is 0.300 gallons per ton of soybeans, and the accountable solvent loss with properly sized, latest technology equipment operating consistently is 0.147 gallons per ton of soybeans, then the remaining "miscellaneous" solvent loss is 0.153 gallons per ton of soybeans.

In order to develop a plan to further improve solvent recovery, and thus minimize solvent loss, we can now apply quality tools and techniques. To begin this process, a Pareto diagram can be effectively used to illustrate the sources of solvent loss and their relative magnitude (Figure 1).

From the Pareto diagram, the primary source of solvent loss in a soybean solvent extraction plant is miscellaneous loss, assuming 0.300 gallons of solvent loss per ton of soybeans and properly sized, latest technology equipment operating consistently. To better identify this miscellaneous solvent loss, such losses can be broken down into three categories:

- A. Excess loss
- B. Fugitive loss
- C. Purging loss

The relative magnitude of each of these loss categories is difficult to measure, but we know that together this miscellaneous loss is the most significant source of solvent loss remaining, and the source that must be managed through a quality process in order to further improve solvent

recovery, and thus minimize solvent loss. A general definition, analysis and action plan for the three categories of miscellaneous solvent loss follows.

#### Excess loss

Excess loss is the amount of additional loss through air from the meal dryer cooler, through meal from the meal dryer cooler, through air from the mineral oil system, through oil from the final oil stripper and through water from the waste water evaporator, as a result of inconsistent operation or a lack of properly sized, latest technology equipment. The excess loss due to inconsistent operation occurs during the period starting when the input conditions change and ending when the input conditions change back to normal, or during the period starting when the input conditions change and ending when steady state is achieved after the operating parameters have been changed.

Examples of excess solvent loss are many. One example is that flake thickness is reduced from 0.014 inches to 0.012 inches. This results in a reduction in extractor drainage rate, which in turn increases the solvent flow rate to the desolventizer toaster. There is a time period in which the solvent flow rate to the desolventizer toaster is increased before temperatures decrease and cause the feedback steam flow controller to add additional steam. During this period of time, the meal discharge temperature is lower than normal and the amount of solvent exiting the desolventizer toaster may increase dramatically. This subse-

quently increases the amount of solvent loss through the air from the meal dryer cooler.

Other examples of excess solvent loss can be more subtle. Solvent is pumped into the work tank from storage. The increased work tank level reduces the total pump head slightly, and therefore the solvent pump flow rate increases slightly. Here solvent is pumped into the extractor, and thus more solvent goes out with the full miscella. This results in lower temperatures and concentrations into the final oil stripper, until the feedback control valve on the second stage evaporator reacts to the change in temperature. During this time period, the amount of solvent in the oil exiting the final oil stripper may increase substantially.

The actions that can be taken to minimize excess solvent loss are:

- Install properly sized, latest technology equipment.
- Improve the consistency of the input product to the solvent extraction plant. This includes rate, moisture, flake thickness, hull content and fines content. It is important to note that this is the responsibility of the soybean receiving and preparation plants, whose internal customer is the soybean solvent extraction plant.
- Improve the consistency of parameters within the solvent extraction plant, particularly during shift changes. This includes levels, pressures, temperatures and flows.
- Install automatic control mechanisms that quickly react to operational inconsistencies and respond by changing operating parameters. It is important to note that these controls must be properly applied to prevent large swings in operating parameters that can cause further inconsistency and excess solvent loss.

#### Fugitive loss

Fugitive loss is the amount of solvent loss from the process equipment through flanges, doors, packing glands, pump seals, valves stems, sight glasses, etc. This loss occurs when the pressure inside of vessels is greater than atmospheric pressure, causing solvent vapor inside the vessel to leak out through any orifice.



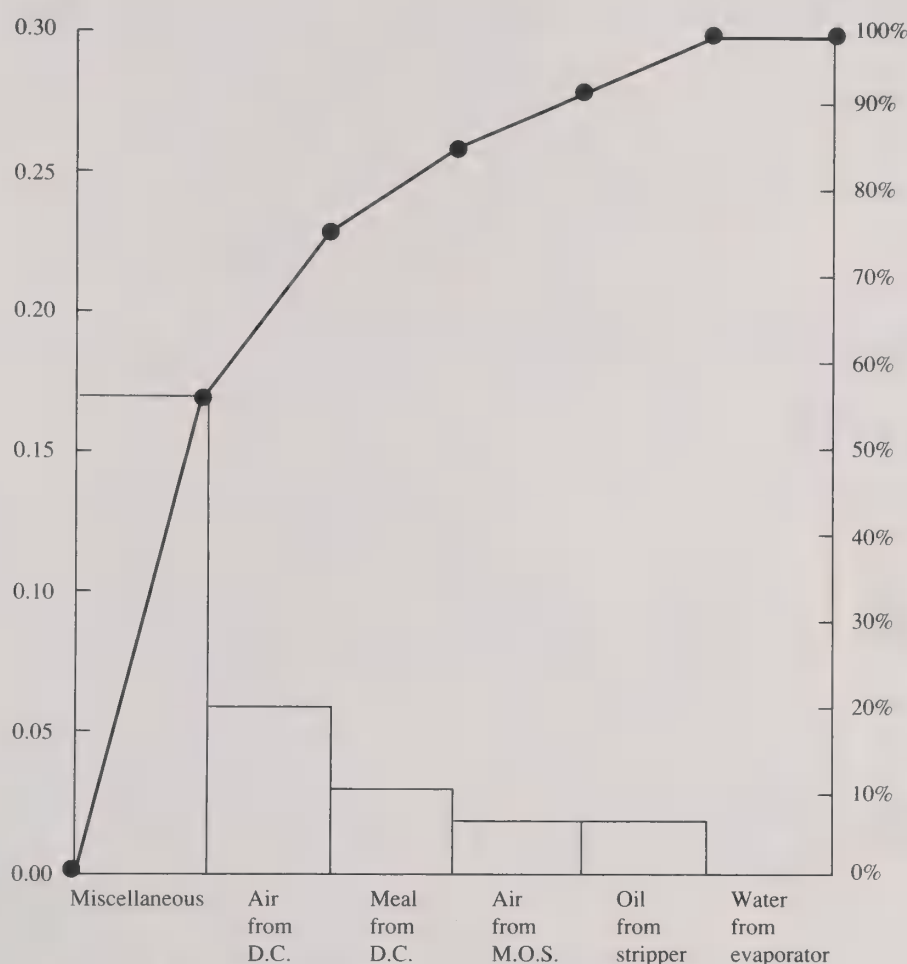


Figure 1. Solvent loss (in gallons solvent/ton of soybeans); D.C.=dryer cooler; M.O.S.= mineral oil system

Fugitive solvent loss is not proportional to rate; therefore, larger plants can be expected to have lower fugitive solvent loss than an average-sized plant, and smaller plants can be expected to have higher fugitive solvent loss than an average-sized plant, on a gallons-per-ton basis.

The actions that can be taken to minimize fugitive solvent loss are:

- Pressure test all equipment and piping with compressed air to ensure that the system is free of leaks prior to starting operation.
- Maintain process equipment under a slight vacuum where possible. This will allow air to leak in, rather than allowing solvent vapor to leak out. It is important to note that too much vacuum will cause excess air to pull in and overload the mineral oil systems, which will cause excess solvent loss.
- Use specially designed seals on any process equipment normally oper-

ating under pressure, where solvent is present. Examples of process equipment normally operating under pressure where solvent is present are miscella pump seals and valve stems, and lower sections of the desolventizer toaster. Double pump seals, special valve stem packings and heavy-duty manway doors with graphite packing are examples of specially designed seals.

#### Purging loss

Purging loss is the amount of solvent loss from the process equipment resulting from freeing the process equipment of solvent vapor for inspection or maintenance. This loss occurs as a result of opening up the process equipment, and as a result of using purge fans to pull air through the process equipment.

During normal operation, purging loss does not exist. Purging loss only exists when the plant is shut down.

Thus, the solvent loss as a result of purging can not be quantified in terms of gallons of solvent loss per ton of soybean processed on a direct basis, as with the other categories of solvent loss. Therefore, purging loss is typically added to total solvent loss on an annual basis, and then factored out in terms of gallons of solvent loss per ton of soybeans processed. When comparing solvent loss from facility to facility, it is important to note whether or not purging loss has been taken into consideration.

The actions that can be taken to minimize purging solvent loss are:

- Improve equipment reliability through design improvement, and through thorough preventative maintenance processes. This will reduce the frequency for which maintenance and inspection are required, thus reducing the frequency of vapor freeing and its related purging solvent loss.
- Allow the normal vapor recovery system to run as long as possible prior to opening up the process equipment. This will recover the majority of the solvent prior to vapor freeing, thus reducing purging solvent loss.
- Discharge the purge fan through a condenser to recover as much as the solvent vapor as possible, thus reducing purging solvent loss.

In summary, with a good solvent loss of 0.300 gallons per ton of soybeans, and with properly sized, latest technology equipment operating consistently, the primary source of solvent loss is the collective miscellaneous loss from excess loss, fugitive loss and purging loss. For the oilseed processing industry to continuously improve solvent recovery, and thus minimize solvent loss, there must be a good quality process in place. A quality process for managing solvent loss is to define each category of loss, collect data and analyze the relative magnitude of each category of loss, select the most significant category of loss, plan actions to minimize this category of loss, execute those actions, evaluate their effect, implement the successful actions, and continuously improve by repeating this quality process. Facilities with good quality processes in place will lead the industry in solvent recovery and minimizing solvent loss.



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*National Emission Standards for Hazardous Air  
Pollutants for Vegetable Oil Production  
Facilities*

**James F. Durham**

Environmental Protection Agency  
Research Triangle Park, NC

# 46th Oilseed Conference

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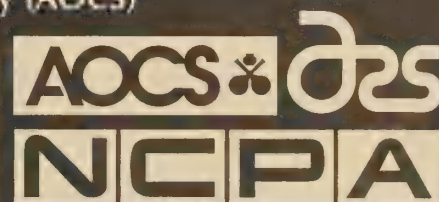
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NATIONAL EMISSION STANDARDS FOR HAZARDOUS AIR POLLUTANTS  
FOR VEGETABLE OIL PRODUCTION FACILITIES

46th Oilseed Conference  
March 9-11, 1997  
New Orleans, Louisiana

James F. Durham  
U.S. Environmental Protection Agency  
Office of Air Quality Planning and Standards  
Research Triangle Park, North Carolina

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OVERVIEW OF PRESENTATION

- EPA organization
- Legislative requirements of Section 112 of the Clean Air Act
- Industry questionnaire
- Next steps in developing standards

## U.S. ENVIRONMENTAL PROTECTION AGENCY

- Administrator
- Administration and Resources Management
- *Air and Radiation*
- Enforcement and Compliance Assurance
- General Counsel
- Inspector General
- International Activities
- Policy, Planning and Evaluation
- Prevention, Pesticides, and Toxic Substances
- Research and Development
- Solid Waste and Emergency Response
- Water
- Regional Offices (1 through 10)

## OFFICE OF AIR AND RADIATION

- *Office of Air Quality Planning and Standards*
  - Office of Atmospheric Programs
  - Office of Mobile Sources
  - Office of Radiation and Indoor Air
- 

## OFFICE OF AIR QUALITY PLANNING AND STANDARDS

- Office of the Director
- Emissions, Monitoring, and Analysis Division
- *Emission Standards Division*
- Air Quality Strategies and Standards Division
- Information Transfer & Program Integration Division



CLEAN AIR ACT - SECTION 112  
HAZARDOUS AIR POLLUTANTS (HAPs)

- Maximum Achievable Control Technology (MACT Standards) or National Emission Standards for Hazardous Air Pollutants (NESHAPS)
  - Section 112(a) - Definitions
    - ◆ Major source - 10 tons/yr any HAP or 25 tons/yr of total HAPs
    - ◆ Area source - Sources that are not major sources
  - Section 112(b) - List of pollutants
    - ◆ Hexane listed as a HAP
  - Section 112(c) - List of source categories
    - ◆ EPA listed Vegetable Oil Production as a source category for final rule in November 2000. [58 FR 83941, December 3, 1993]
- 

CLEAN AIR ACT - SECTION 112  
HAZARDOUS AIR POLLUTANTS (HAPs)  
(Continued)

- Section 112(d) - Emission standards
  - ◆ Existing sources - Not to be less stringent than the average of the best performing 12 percent of existing sources. This is termed "The Floor".
  - ◆ New sources - Not less stringent than the best performing existing source
  - ◆ Source categories can be divided into subcategories with similar characteristics



## FORMAT OF REGULATIONS

- Specific requirements for each emission point
  - ◆ Each process vent
  - ◆ Solvent transfer and storage
  - ◆ Waste water collection and treatment
  - ◆ Equipment leaks (valve & pump seals, flanges ...)
- Overall plant performance standard (gal/ton)
  - Preferred approach by industry and regulatory agencies
  - ◆ Provides industry flexibility to implement cost-effective approaches
  - ◆ Minimizes monitoring record keeping and reporting
  - ◆ Provides incentive to reduce loss through process changes



## QUESTIONNAIRE

- Distributed in June 1996 after consultation and assistance from the Vegetable Oil MACT Coalition
  - Vegetable Oil MACT Coalition
    - ◆ Corn Refiners Association
    - ◆ National Cotton Council
    - ◆ National Cottonseed Products Association
    - ◆ National Peanut Council
    - ◆ National Oilseed Processors Association
  - Sent to 32 companies requesting information on 112 solvent extraction plants
    - ◆ Products, production rates, seed processing rate, number of employees
    - ◆ Gallons of solvent used per ton of seeds crushed for each month in 1995
    - ◆ Type and age of process equipment
    - ◆ Identify points of solvent emissions to the atmosphere and emission controls
- 

## SUMMARY OF QUESTIONNAIRE RESPONSES

- 105 facilities submitted complete responses
  - ◆ 64 soybean
  - ◆ 25 cottonseed
  - ◆ 31 other
- Approximately 70% of the responses included "Confidential Business Information"

## RELATIVE MAGNITUDE OF EMISSIONS

Source Category	Current Emissions - (1000s ton/year)	
	VOC	HAP
Synthetic Organic Chemicals	1,300	570
Petroleum Refineries	460	90
Vegetable Oil	44	28
Gasoline distribution	280	18
Marine Terminals	43	4.5

- The lowest hexane emissions reported from one plant were 65 tons/year. Therefore, it appears that all plants exceed the 10/25 HAP cutoffs in Section 112(a) of the CAA and would be subject to the regulations.

## OILSEED PROCESSED AND SOLVENT USED IN 1995

Oilseed	Seed Processed (million tons)	Solvent Use	
		(million gal)	(gal/ton)
Soybean	41.8	11.6	0.28
Cottonseed	3.9	2.5	0.64
Other	4.9	1.7	0.35

# SOLVENT LOSSES - AVERAGE OF TOP 12 PERCENT

Type of Seed	Average of top 12% (gal/ton)
All oilseeds	0.16
Soybeans only	0.16
All seeds other than soybeans	0.20

Based average of monthly values of seeds crushed and solvent used that were reported in 1995.

## REDUCTION IN SOLVENT LOSSES

1000's tons seeds crushed in 1995	Number plants		% Reduction to meet average Top 12 percent
	Soybean	Other	
<20	0	4	82
20-50	0	8	79
50-100	0	11	79
100-150	0	11	71
150-200	2	6	60
200-300	3	5	42
300-400	5	3	46
400-500	6	2	52
500-600	7	1	39
600-700	12	0	38
700-800	7	0	37
800-900	7	1	16
>900	10	0	39

## NEXT STEPS IN REGULATORY PROCESS

- Meet with individual companies to further our understanding of solvent losses
    - ✦ Identify what the efficient plants are doing to minimize losses
    - ✦ Changes the less efficient plants must make to reduce solvent losses
    - ◆ The costs associated with reducing solvent losses
    - ✦ Factors that may limit certain types of plants from improving recovery efficiency
- 

## NEXT STEPS IN REGULATORY PROCESS (Continued)

- Form stakeholder group - industry, State/local agencies and EPA
- Determine and analyze regulatory options
  - ◆ Subcategory determination
  - ✦ Determine the minimum control level (floor) for each subcategory
  - ◆ Determine the environmental and economic impacts of compliance
  - ✦ Prepare regulatory recommendations



*Proposed Changes to the NAAQS  
for PM and Ozone*

**Pat Delamater**  
Trinity Consultants Incorporated  
Dallas, TX

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# PROPOSED CHANGES TO THE NAAQS FOR PM AND OZONE

## Introduction

The United States Environmental Protection Agency (U.S. EPA) is proposing significant changes to the National Ambient Air Quality Standards (NAAQS) for particulate matter and ozone. Since formally announced on November 27, 1996, this proposed action has made national headlines as being the most significant air quality legislation since passage of the 1990 Clean Air Act Amendments. The new standards will significantly increase the number of ozone and particulate matter nonattainment areas across the country, resulting in more stringent requirements for new facilities and those facilities that plan future production increases.

Industry groups are aggressively lobbying against the U.S. EPA proposal, stating that the cost to demonstrate compliance with the new standards would be billions of dollars, with no significant health benefits realized. At the same time, environmentalist groups and public health advocates argue that the more stringent regulations are necessary to meet the goals of the Clean Air Act and protect the public health.

The proposed standards will affect facilities that emit two criteria air pollutants: particulate matter 2.5 microns and less ( $PM_{2.5}$ ) and volatile organic compounds (VOCs), such as hexane. Obviously, the implications of these new standards on the cottonseed oil mill industry are significant. Historically, cottonseed oil mills have been located in rural areas, some distance from the more polluted urban cities that have been designated as not attaining the NAAQS. Only a hand-full of mills have had to deal with the more stringent air permitting rules that pertain to nonattainment areas. If the new standards are promulgated as proposed, the number of nonattainment areas will increase dramatically and a greater number of cottonseed oil mills will be challenged with addressing these new standards while still attempting to maximize production potential.

This paper provides a summary of the recent events leading up to U.S. EPA's new NAAQS proposals. An historical review of the development of the current NAAQS for particulate matter and ozone is provided along with the legal basis for U.S. EPA to review and revise the standards. This paper also outlines the substantive content of the proposed revisions as compared to the

existing NAAQS and identifies those areas of the country that will likely be affected. Finally, a discussion of how political lobbies could potentially affect the proposed rule is presented along with guidance on how the cottonseed oil mill industry should begin preparing for the eventual affects of the proposed standards.

### **Chronology of Events Leading to the Proposed Regulations**

The last review of particulate matter air quality criteria and standards was completed in July 1987.<sup>1</sup> At that time, U.S. EPA decided to modify the particulate matter indicator from total suspended particulate (TSP) to particulate matter 10 microns and less (PM<sub>10</sub>). As directed by the Clean Air Act to review each NAAQS every five years, U.S. EPA formally initiated the current review of the standard in April 1994 by announcing its intentions to develop a new Criteria Document for particulate matter.<sup>2</sup>

The impending revisions to the particulate matter NAAQS were accelerated pursuant to a court-ordered deadline set in the case *American Lung Association (ALA) v. Browner* (U.S. District Court of Arizona, No. 93-643) on October 6, 1994.<sup>3</sup> Several workshops were held in November 1994 and January 1995 by U.S. EPA's National Center for Environmental Assessment (NSEA) to discuss health-based criteria that would be used to establish the new standard. The conglomeration of this new health-based information was documented in what U.S. EPA refers to as a Criteria Document. External review drafts of the Criteria Document were made available for public comment and were reviewed by the Clean Air Scientific Advisory Committee (CASAC) in August and December 1995. CASAC is a standing committee of U.S. EPA's Science Advisory Board established under Section 109(d)(2) of the Clean Air Act.

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<sup>1</sup> U.S. EPA, Office of Air Quality Planning and Standards, *Review of the National Ambient Air Quality Standards for Particulate Matter: Policy Assessment of Scientific and Technical Information*, (Research Triangle Park, NC: U.S. EPA EPA-452/R-96-013, July 1996), p. II-3.

<sup>2</sup> U.S. EPA, 40 CFR Part 50, *National Ambient Air Quality Standards for Particulate Matter: Proposed Decision*, December 13, 1996, p. 15.

<sup>3</sup> Court Decision: *American Lung Association v. Browner* (CIV-93-643-TVC-ACM), D. Ariz., October 6, 1994.



On March 15, 1996, CASAC issued a closure letter to U.S. EPA regarding the Criteria Document which allowed U.S. EPA to move forward in the development process.<sup>4</sup> In July 1996, a final Staff Paper was released.<sup>5</sup> The Staff Paper evaluates policy implication of the key studies and scientific information contained within the Criteria Document, identifies critical elements that U.S. EPA staff believe should be considered, and presents U.S. EPA staff conclusions and recommendations of suggested options for the Administrator's consideration. In short, the Staff Paper contains the scientific basis for U.S. EPA's proposed changes to the NAAQS.

On November 27, 1996, U.S. EPA formally announced their proposal to revise the NAAQS for particulate matter and ozone. The proposal was documented in the December 13, 1996 *Federal Register*.<sup>6</sup> Public meetings were held on January 14-15, 1997 in Salt Lake City, Utah and Research Triangle Park, North Carolina. In late January 1997, under significant pressure from industry groups and governors, U.S. EPA Administrator Carol Browner agreed to request a 60-day extension to the public comment period and the promulgation date. However, on February 10, 1997, the Arizona District Court denied U.S. EPA's request for a 60-day extension. Although the court rejected the agency's petition for a two-month extension, the judge did grant an extension of three weeks. Therefore, the public comment period will close March 12, 1997 and the publication of the final decision of the rules will be July 19, 1997.<sup>7</sup> The court stated that there would be no further extension granted.

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<sup>4</sup> U.S. EPA, *Review of the National Ambient Air Quality Standards for Particulate Matter*, Appendix H (Closure Letter dated March 15, 1996).

<sup>5</sup> U.S. EPA, *Review of the National Ambient Air Quality Standards for Particulate Matter*, OAQPS Staff Paper, EPA-452/R-96-013, July 1996.

<sup>6</sup> U.S. EPA, *Federal Register, National Ambient Air Quality Standards for Particulate Matter: Proposed Decision*, December 13, 1996.

<sup>7</sup> U.S. EPA, *Extension of public comment period and announcement of availability of additional reports*, February 10, 1997.

In August 1994, U.S. EPA initiated its review of the air quality criteria and standards for tropospheric ozone and related photochemical oxidants on public health and welfare.<sup>8</sup>

Tropospheric ozone is chemically identical to stratospheric ozone, which is produced miles above the earth's surface and provides a protective shield from excess ultraviolet radiation. In contrast, excessive concentrations of tropospheric ozone have been associated with harmful effects due to its oxidative properties in the presence of people and plants during respiratory processes. Ozone formation is the result of chemical reactions of VOCs, nitrogen oxides (NO<sub>x</sub>), and oxygen in the presence of sunlight. Although revisions to the ozone standard are not under a court-ordered deadline, the development process of the revised standard has occurred simultaneously with the review for particulate matter. According to U.S. EPA, the criteria reviews, along with related implementation strategy activities, have brought forth an important linkage between fine particulate matter and ozone.<sup>9</sup> A summary of the chronology of these new standards compiled by the U.S. EPA is contained in Figures 1 and 2 for particulate matter and ozone, respectively.

### **Historical Overview of the Particulate Matter and Ozone NAAQS**

The origin of the air quality rules and regulations that we have today is the 1967 Air Quality Act. Following passage of this Act, the 1970 Clean Air Act Amendments (CAAA) "put teeth" into the Clean Air Act. The goal of the 1970 amendments was "to protect and enhance the quality of the nation's air resources so as to promote public health and welfare and the productive capacity of its population."<sup>10</sup> The most significant provisions of the 1970 CAAA were the development of NAAQS and the designation of nonattainment areas.

The NAAQS are "health-based" standards set at levels allowing an adequate margin of safety required to protect the most sensitive individuals. The margin of safety requirement for these

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<sup>8</sup> U.S. EPA, Federal Register, *National Ambient Air Quality Standards for Ozone: Proposed Decision*, December 13, 1996

<sup>9</sup> U.S. EPA, Federal Register, *National Ambient Air Quality Standards for Ozone: Proposed Decision*, p. 17, December 13, 1996

<sup>10</sup> U.S. Congress, *The Clean Air Act as Amended August 1977*, 95th Cong., 1977 (Washington, D.C.: GPO, Public Law 95-11, 1977).

Figure 1. Chronology of Particulate Matter NAAQS Revision Process

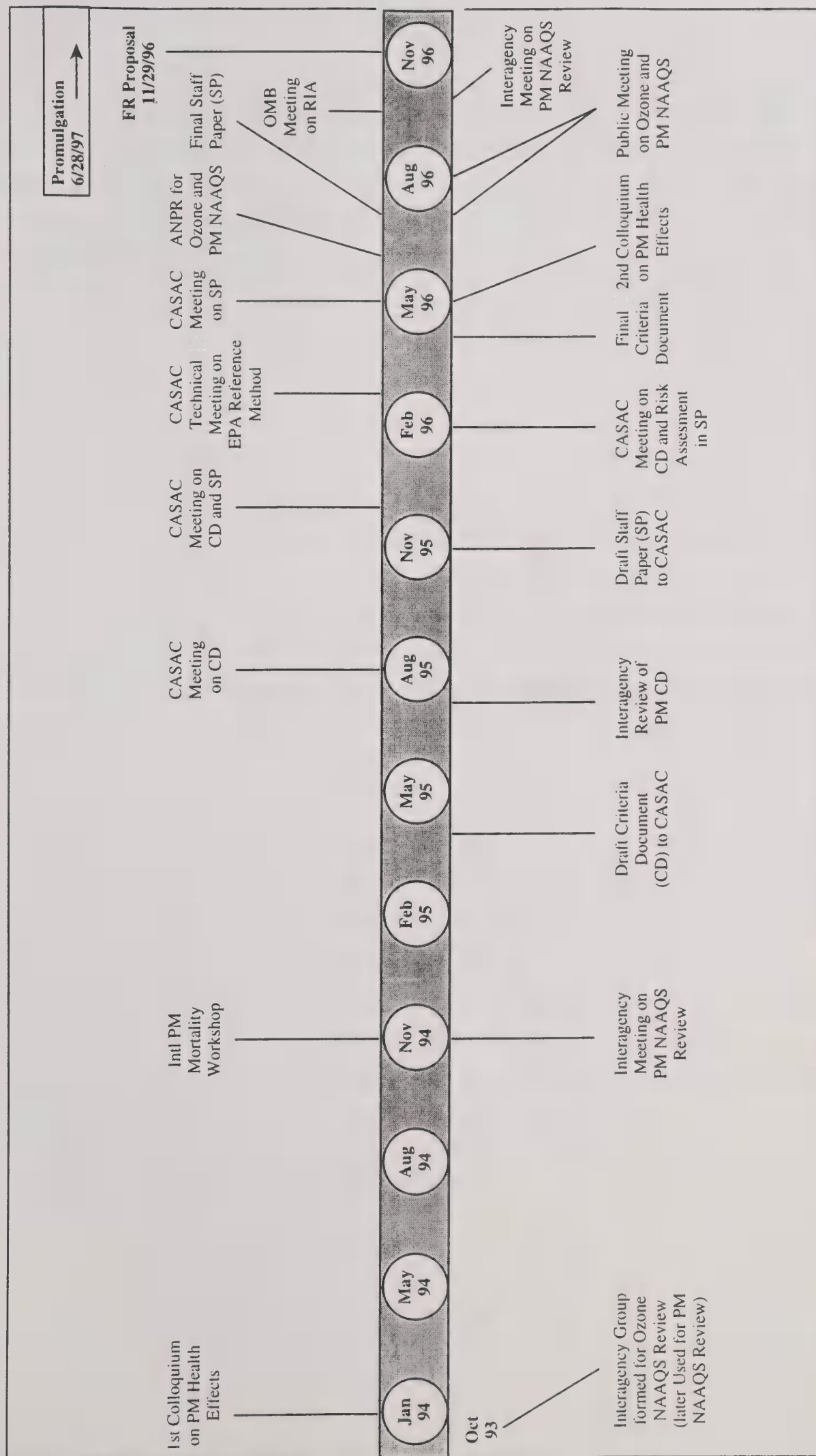
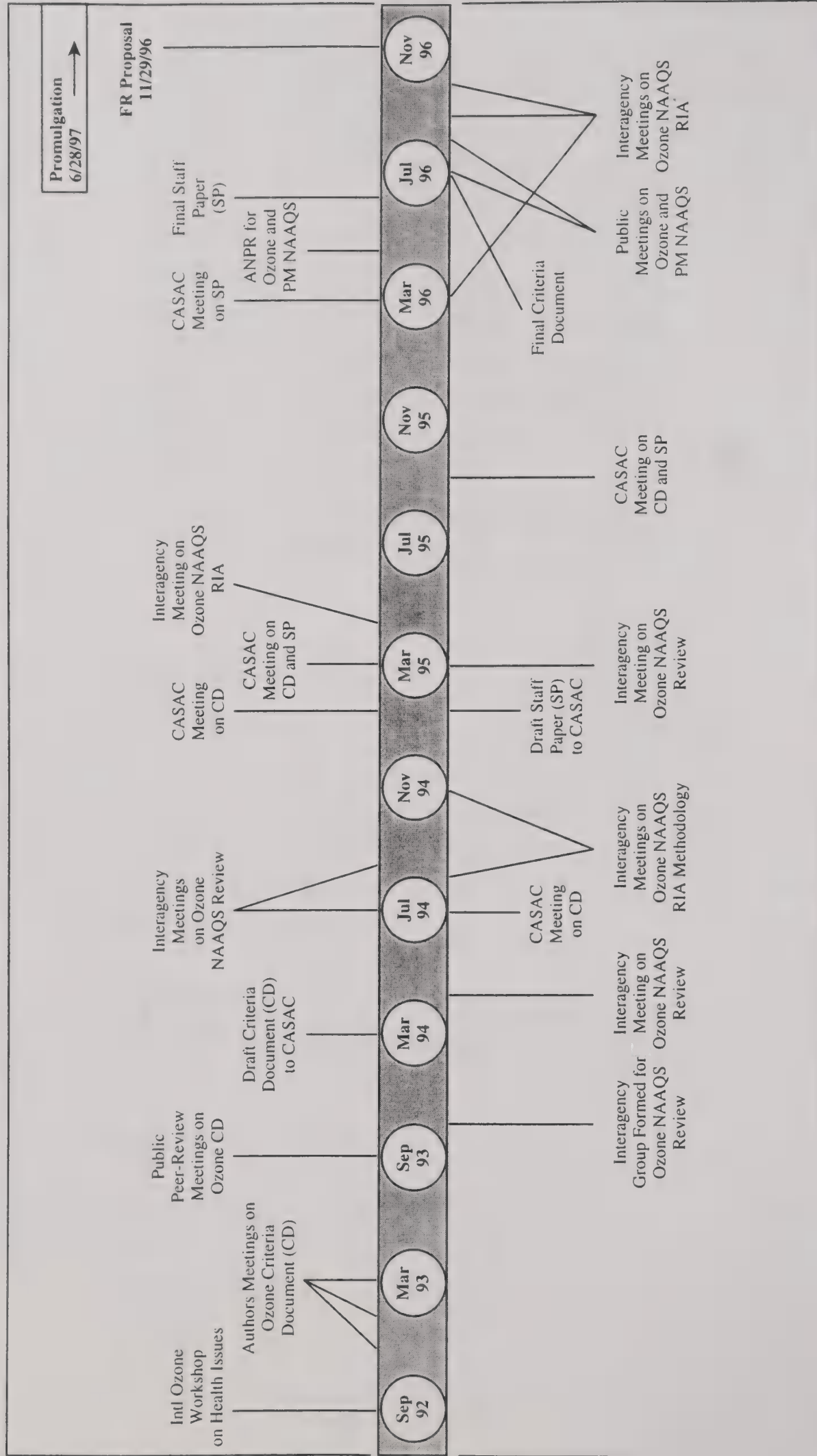


Figure 2. Chronology of Ozone NAAQS Revision Process





“primary standards” is intended to address uncertainties associated with inconclusive scientific and technical information available at the time the standard is set and to provide a reasonable degree of protection against hazards that research has not yet identified. In addition, “secondary standards” are established to protect the “public welfare” from any known or anticipated adverse effects associated with the presence of a pollutant in the ambient air. Welfare effects include, but are not limited to, soils, water, crops, vegetation, animals, and visibility. NAAQS are established for particulate matter, nitrogen dioxide, sulfur dioxide, carbon monoxide, ozone, and lead.

In addition to the development of NAAQS, the 1970 CAAA classified certain areas of the country based on the existing level of pollution in that area. If air pollution concentrations exceeded the NAAQS, the area was to be designated as nonattainment, or not attaining the NAAQS. The Houston, Dallas/Fort Worth, Chicago, Los Angeles, and Atlanta metropolitan areas are examples of nonattainment areas for ozone. Facilities that operate in nonattainment areas must deal with more stringent air quality criteria when modifying their operation. Due to the more complex permitting process, these more stringent regulations may place facilities that operate in nonattainment areas at a competitive disadvantage with those that operate in attainment areas.

Since 1970, the U.S. EPA has been mandated by two sections of the Clean Air Act to establish and periodically review the NAAQS. Section 108 directs the Administrator to identify pollutants which “may reasonably be anticipated to endanger public health and welfare” and to establish standards that will address these concerns.<sup>11</sup> The standards are to “accurately reflect the latest scientific knowledge useful in indicating the kind and extent of all identifiable effects on public health and welfare.” Section 109 directs the Administrator to propose and promulgate NAAQS for each pollutant identified under Section 108. Section 109 also requires periodic review, and, if appropriate, revision of existing air quality criteria and NAAQS.<sup>12</sup>

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<sup>11</sup> U.S. Congress, *The Clean Air Act as Amended August 1977*, pp. 12-15. <sup>11</sup> U.S. Congress, *The Clean Air Act as Amended August 1977*, pp. 15-17.

<sup>12</sup> U.S. Congress, *The Clean Air Act as Amended August 1977*, pp. 15-17.

## Summary of Proposed NAAQS Revisions

Changes to the particulate matter and ozone standards are being proposed because U.S. EPA feels that the public is at significant risk even when pollutant concentrations are less than the existing standards. U.S. EPA Administrator Browner has stated that “this is one of the most significant decisions I will make to protect public health and welfare in this country. We believe very, very strongly that there are real and significant health problems from this kind of pollution and that our final decision must be standards that protect against those.”<sup>13</sup> The following subsections review the substantive content of the proposed revisions.

### Particulate Matter

The proposed revisions to the particulate matter NAAQS are the result of an extensive review of thousands of scientific studies that highlight over 80 key epidemiological studies. Over 60 of these epidemiological studies have determined significant links between particulate matter levels at or below the current standards and premature death and illness.<sup>14</sup> The studies have indicated that fine and coarse particulate matter are fundamentally two different pollutants: PM<sub>10</sub> and PM<sub>2.5</sub>. Of the 21 CASAC panel members, 19 have recommended revising the current PM<sub>10</sub> standards by adding standards for fine particulates (PM<sub>2.5</sub>).<sup>15</sup>

The Criteria Document states that the recent epidemiological studies provide “evidence that serious health effects are associated with exposure to ambient levels of particulate matter found in contemporary urban airsheds even at concentrations below the current

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<sup>13</sup> “Too Strict or Not Strict Enough?”, *EM*, January 1997; pp. 16-17.

<sup>14</sup> U.S. EPA, *Review of the National Ambient Air Quality Standards for Particulate Matter*, Chapter V.

<sup>15</sup> Air & Waste Management Association, *Proposed Revisions to the Ozone and Particulate Matter National Ambient Air Quality Standards (Roundtable Discussion, Irving, TX)*, February 11, 1997.

standards.”<sup>16</sup> These studies were conducted in the urban airsheds of Los Angeles, California and Philadelphia, Pennsylvania. The key health effects categories associated with PM<sub>2.5</sub> exposure include:

- Premature mortality
- Aggravation of respiratory and cardiovascular disease
- Changes in lung function and increased respiratory symptoms
- Changes to lung tissues and structure
- Altered respiratory defense mechanisms

The current PM<sub>10</sub> standards are 50 micrograms per cubic meter (µg/m<sup>3</sup>) based on an annual arithmetic mean (averaged over three years) and 150 µg/m<sup>3</sup> based on a 24-hour average (averaged over three years), with one expected exceedance per year. PM<sub>10</sub> is roughly one-third the width of a human hair and is mainly composed of dust, pollen, fly ash, and suspended soils.

The proposed PM<sub>2.5</sub> standards are 15 µg/m<sup>3</sup> based on an annual arithmetic mean, spatial average of designated monitors (averaged over three years), and 50 µg/m<sup>3</sup> based on a 24-hour average, 98th percentile concentration, maximum monitor in an area (averaged over three years). The 98th percentile concentration is the eighth highest 24-hour concentration occurring within a year assuming 365 days of data are considered. PM<sub>2.5</sub> is mainly composed of products of combustion, waste streams off high-efficiency dust collectors, soot, aerosols, sulfates and nitrates and can be generated via chemical reactions and condensation from gases.

The U.S. EPA is also proposing to revise the current 24-hour primary PM<sub>10</sub> standard of 150 µg/m<sup>3</sup> by replacing the 1-expected-exceedance form with a 98th percentile form, averaged over 3 years at each monitor within an area. U.S. EPA is soliciting comment on an alternative proposal that would revoke the 24-hour PM<sub>10</sub> standard. U.S. EPA is

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<sup>16</sup> U.S. EPA, Federal Register, *National Ambient Air Quality Standards for Particulate Matter: Proposed Decision*, December 13, 1996.

proposing to retain the current annual primary PM<sub>10</sub> standard of 50 µg/m<sup>3</sup>. The secondary standards are being proposed to be made identical to the proposed primary standards.

According to U.S. EPA, the revised particulate matter standards will result in:

- Approximately 20,000 to 40,000 fewer premature deaths per year, especially to the elderly and those with heart and lung disease
- Approximately 10,000 fewer respiratory-related hospital admissions per year
- Hundreds of thousands fewer incidences each year of aggravated asthma and respiratory symptoms
- Tens of thousands fewer cases each year of chronic bronchitis
- Reduced risks of more frequent childhood illnesses, which are of concern both in the short-term as well as for the future development of healthy lungs in affected children

## Ozone

As with particulate matter, extensive review of thousands of scientific studies highlighted over 180 key health effects studies as the basis for revising the ozone NAAQS. These studies indicated that ozone levels less than the current standard would cause significant health effects in children and other susceptible groups, which make up over one-third of the population. CASAC unanimously recommended that U.S. EPA replace the existing 1-hour ozone standard with a new 8-hour standard to protect against longer exposures that have now been clearly related to health effects at lower concentrations under typical exposure conditions.<sup>17</sup> The key health effects categories associated with ozone exposure include:

- Moderate decreases in lung function
- Respiratory symptoms such as those associated with chronic bronchitis

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<sup>17</sup> Air & Waste Management Association, *Proposed Revisions to the Ozone and Particulate Matter National Ambient Air Quality Standards (Roundtable Discussion, Irving, TX)*, February 11, 1997.



- Increase respiratory problems resulting in increased hospital admissions
- Chronic inflammation and irreversible structural damage in the lungs
- Growing evidence suggests association with premature death

The current primary ozone standard is 0.12 parts per million (ppm) based on a 1-hour average, no more than three exceedances over three consecutive years. Under the current proposal, attainment of standards would no longer be based upon 1-hour averages, but on 8-hour averages. Due to change in the averaging period, U.S. EPA is proposing to lower the standard from the present 0.12 ppm to 0.08 ppm (based on conventional rounding, i.e., 0.084 ~ 0.08). The proposed primary standard would be based on a 3-year average of the third highest daily maximum 8-hour ozone concentration.

The current secondary standards may also be modified using either the primary standard or a new standard based on the seasonal “SUM06” approach. The SUM06 approach is expressed as a sum of hourly ozone concentrations greater than or equal to 0.060 ppm, summed over 12 hours per day during the 3-month period when ozone concentrations are at their highest. Such a standard would not be attained when the sum exceeds 25 ppm-hour.

According to U.S. EPA, the revised ozone standards will result in:

- Approximately one million fewer incidences of significant decreases in lung function per year in children
- Hundreds of thousands of fewer incidences of moderate to severe respiratory symptoms per year (e.g., aggravated coughs and chest pains)
- Significantly fewer incidences of lung inflammation
- Fewer hospitalizations and emergency room visits for individuals with asthma
- Reduction in premature deaths of the elderly

## Strategies to Comply with Revised NAAQS and Associated Costs

U.S. EPA expects that the revised standards will be generally achieved through programs currently developed or implemented, such as the current Acid Rain Program, inspection / maintenance (I/M) programs in metropolitan areas, and Maximum Achievable Control Technology (MACT) requirements. However, in the more severely polluted areas, additional control measures will likely be necessary, including cleaner burning gasoline and additional control requirements on certain industrial processes.

In the past, the burden of developing a program that would attain and maintain compliance with the NAAQS has been upon the individual states through their State Implementation Plans (SIPs). However, during the past several years, a more meso-scale regional approach has been adopted that evaluates entire regions and should change the way implementation strategies are developed for NAAQS.

The U.S. EPA estimates that the combined cost of implementing and complying with the proposed NAAQS for particulate matter and ozone will be \$8 to \$10 billion. This equates to approximately \$30 per person or \$0.09 per person per day. U.S. EPA estimates the combined monetized benefits associated with the proposed NAAQS for particulate matter and ozone will be \$58 to \$120 billion. Thus, the net benefits to the overall economy will be \$51 to \$112 billion.<sup>18</sup> U.S. EPA feels these cost estimates are overstated for the following reasons:

- U.S. EPA cost estimates have historically been proven to be significantly higher than actual costs
- Air quality models used for national analyses are oversimplified
- U.S. industry tends to rapidly adjust to stricter pollution requirements in innovative and cost effective ways
- Separate cost analyses were conducted for particulate matter and ozone even though significant overlap may exist in both benefits and cost

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<sup>18</sup> Ibid.

Industry representatives feel that the U.S. EPA cost analyses are flawed and greatly under-estimate the cost of implementing the proposed standards. Industry has stated that U.S. EPA's cost data only considers the cost of control equipment / techniques that will be implemented on sources to reduce emissions. However, the cost estimate does not account for the development of SIPs by each affected state agency. Industry has warned that tax revenues must be allocated to support such a significant change in states' air regulations. Both industry and state governments have indicated that the U.S. EPA proposals will result in unfunded federal mandates on states' air permitting programs.

### **Attainability of the Proposed Standards - An Industry Prospective**

Based on several technical publications, the attainability of the proposed standards is highly uncertain. The attainability analyses for the current ozone standards are mostly based on a background hourly ozone concentration of  $40 \mu\text{g}/\text{m}^3$  recommended by the U.S. EPA. This background concentration is employed in the absence of anthropogenic or biogenic emissions of VOC or  $\text{NO}_x$ . This is an important basis for the development of states' ozone attainment strategies. However, in a recent background ozone review performed by U.S. EPA with data collected at six clean sites as background concentrations, the third highest 8-hour daily maximum concentrations averaged over three years (1993-1995) ranged from 45 to 61 parts per billion (ppb). This finding indicates that the background ozone level used for ozone attainability analyses may be too low to provide a realistic basis for future attainment demonstrations.

With regard to the particulate matter NAAQS, although the CASAC review indicated that a new  $\text{PM}_{2.5}$  standard should be established, there is no consensus on the level, averaging time, or form of a  $\text{PM}_{2.5}$  NAAQS. CASAC acknowledged that there are many unanswered questions due to the lack of nationwide  $\text{PM}_{2.5}$  data. A recent particulate matter study indicates that the background "fine" particulate matter concentration is

typically in the range of 15 to 25  $\mu\text{g}/\text{m}^3$ .<sup>19,20</sup> These concentrations are generally lower than the average indoor fine particulate matter concentrations measured in a non-smoking environment. As indicated in a U.S. EPA published review, the particulate matter standard may be well below indoor air concentrations in major cities. Although human activities mostly take place indoors in urbanized areas, U.S. EPA's health-based analyses, including the *Regulatory Impact Analysis*, did not draw any conclusions on the exposure that may be due to indoor dust (primarily contributed by smoking, cooking and other indoor air dust) rather than ambient air concentration. These health-based studies did not provide any baseline concentration for  $\text{PM}_{2.5}$  as a threshold concentration for a risk-free  $\text{PM}_{2.5}$  exposure. The main concern regarding the  $\text{PM}_{2.5}$  NAAQS is how U.S. EPA can demonstrate the standards that are proposed today are attainable since the nation is lacking reliable  $\text{PM}_{2.5}$  ambient data.

### **Areas Likely to be Affected by the Proposed Standards**

Figure 3 shows the areas of the southeast and south-central United States that will be affected by the proposed NAAQS revisions based on U.S. EPA estimates. Figure 4 shows the same information based on an assessment by the American Petroleum Institute (API). The API maps contrast sharply to U.S. EPA's predictions on the number of counties that would violate the proposed standards. According to API, the U.S. EPA maps fail to depict those transitional areas that fall between the concentration-based averages used to determine compliance with the ozone standard. API is concerned that if promulgated, the more stringent ozone standards will subject large portions of the nation to unprecedented regulatory burdens.

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<sup>19</sup> Altshuller, A.P., Lefohn, A.S., Background Ozone in the Planetary Boundary Layer over the United States, *J. Air and Waste Management Association*, 46:134-14, 1996.

<sup>20</sup> Wallace, L., Indoor Particles: A Review, *J. Air and Waste Management Association*, 46:98-126, 1996.



Figure 3. Counties Predicted by U.S. EPA to be in Violation of the Ozone NAAQS

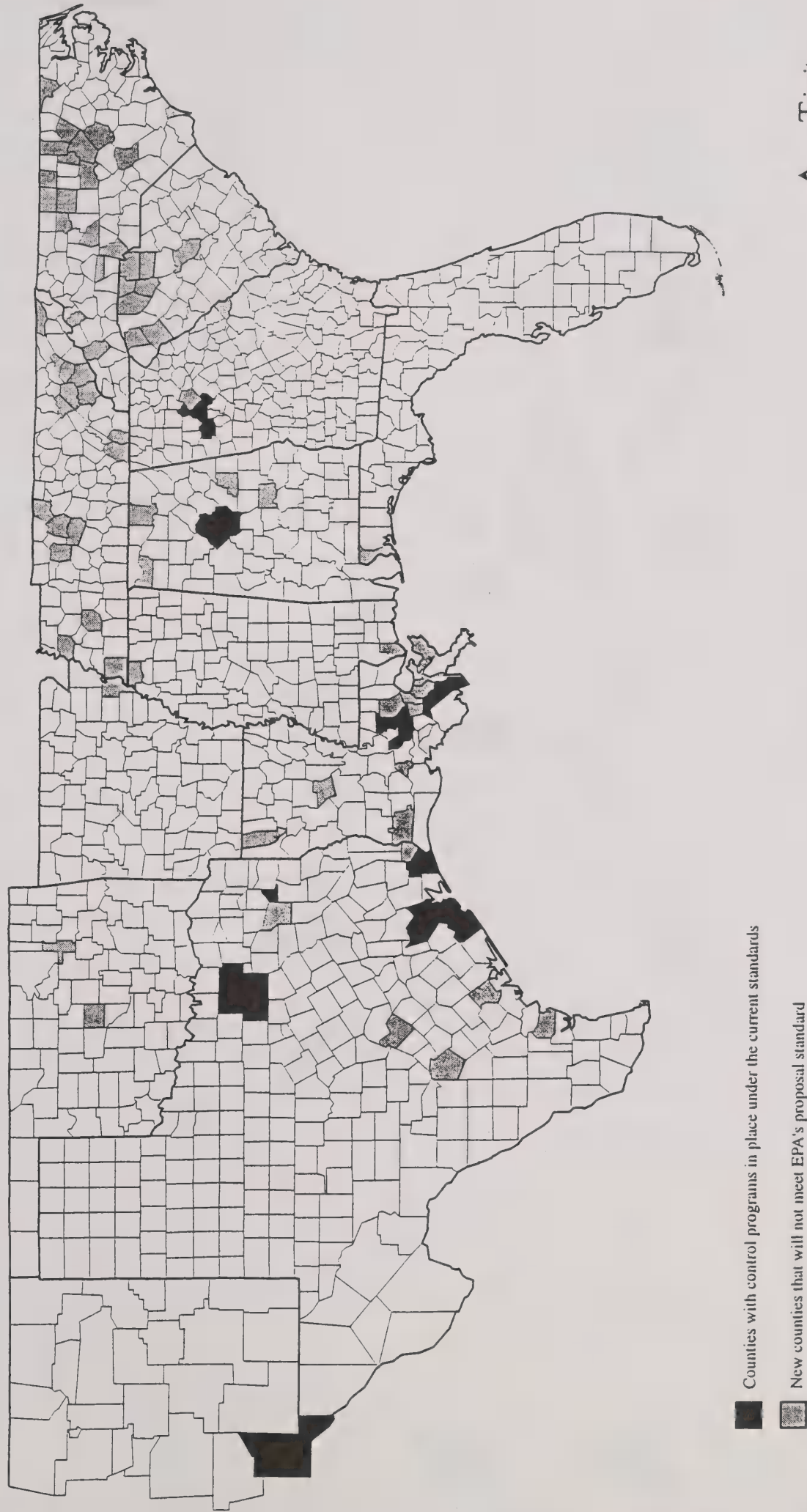
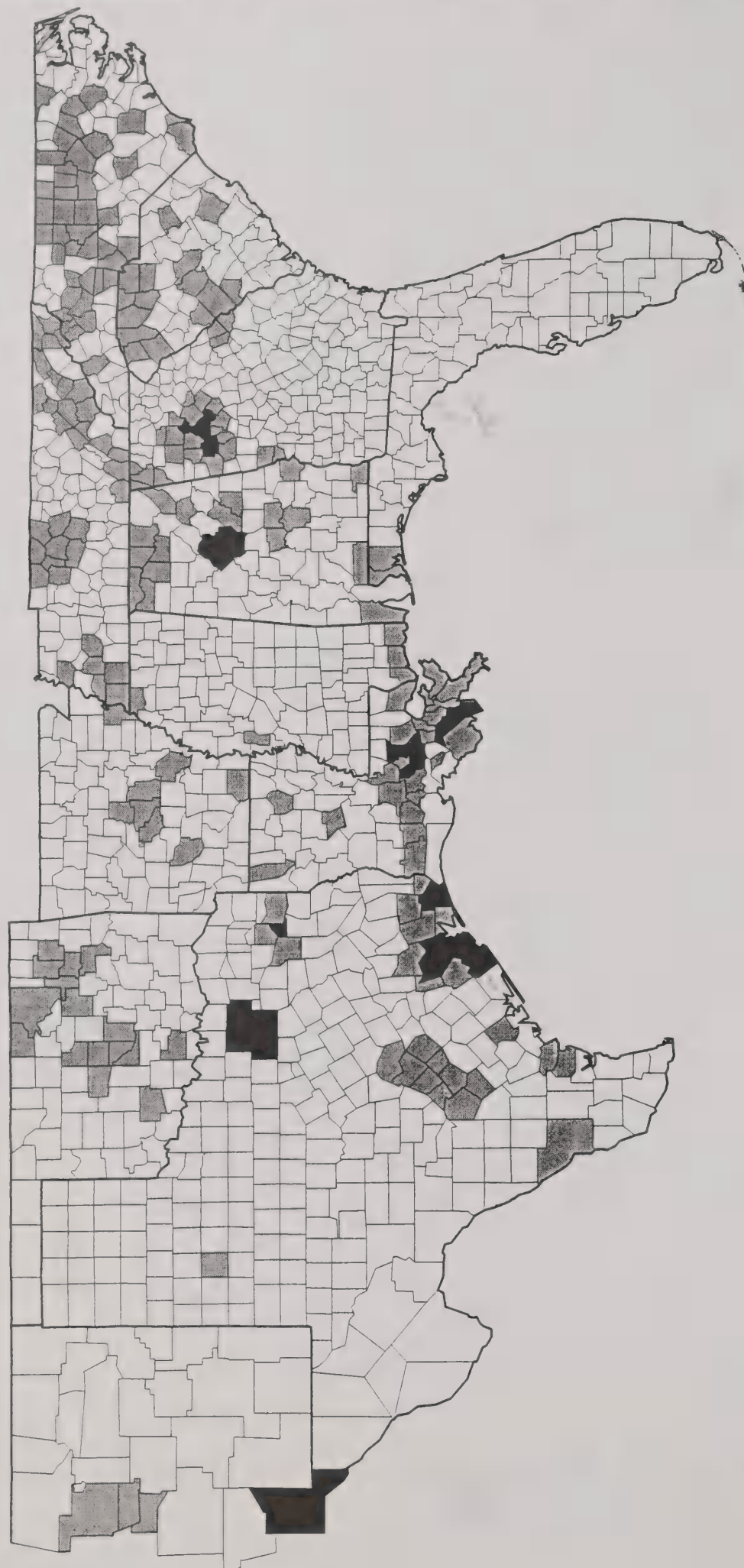


Figure 4. Counties Predicted by API to be in Violation of the Ozone NAAQS



■ Counties with control programs in place under the current standards

■ New counties that will not meet EPA's proposal standard

Source: 1993-1995 data

## **How the Proposed Standards will Affect the Cottonseed Oil Mill Industry**

Historically, cottonseed oil mills have not operated in urban areas that have been designated as not attaining the NAAQS for a particular criteria air pollutant. The only oil mills that currently operate in nonattainment areas are located in Richmond, Texas and Fort Worth, Texas.

However, following promulgation of the proposed standards, facilities that operate in the following areas will be required to adhere to nonattainment regulations (based on the API report):

- Richmond, Texas (Fort Bend County)
- Fort Worth, Texas (Tarrant County)
- Lubbock, Texas (Lubbock County)
- Oklahoma City, Oklahoma (Oklahoma County)
- Little Rock, Arkansas (Pulaski County)
- West Monroe, Louisiana (Ouachita Parish)
- Memphis, Tennessee (Shelby County)
- Corcoran, California (Kings County)

Facilities operating in these areas account for approximately one-third of the oil mills in the country and approximately 40 percent of the total cottonseed processed. For facilities that operate in these areas, relatively small increases in emissions (as low as 25 tons per year) could trigger federal nonattainment new source review permitting requirements. For example, if a facility increases production by 50 tons of cottonseed per day (at a constant hexane disappearance rate of 0.50 gallons per ton) in the Houston nonattainment area, the facility may trigger federal nonattainment permit applicability. These permitting requirements are far more complex and far more costly than those applicable to facilities operating in attainment areas.

Key federal nonattainment permitting requirements include:

- Lowest Achievable Emission Rate (LAER) technology for new and modified units
- Emissions offsets at rates relative to the severity of the nonattainment area
- Demonstration of state-wide compliance
- Demonstration that the modification will not adversely effect nearby Class I areas

Facilities that operate in attainment areas may also be affected by U.S. EPA's proposed changes for  $PM_{2.5}$ . For example, Texas currently requires applicants to demonstrate compliance with the  $PM_{10}$  NAAQS by completing a comprehensive computer air dispersion modeling analysis. It is expected that this requirement will also apply to  $PM_{2.5}$ . Thus, facilities submitting permit applications in Texas will be required to first quantify the  $PM_{2.5}$  fraction of particulate matter emitted from their sources, and second, demonstrate compliance with the  $PM_{2.5}$  NAAQS through an air dispersion modeling analysis. Other states are likely to have similar new source review permitting requirements, even for SIP-type permits.

### **Interim Implementation Policy**

Based on initial guidance from U.S. EPA, an Interim Implementation Policy (IIP) will be established that will continue to enforce existing particulate matter and ozone designations until the new designations are developed based on the new standards. The IIP will be effective from the date that the new standards are promulgated (expected to be July 19, 1997) until the states develop new SIPs that receive approval from U.S. EPA (not expected to be earlier than 2002). The IIP is strongly based on the principle of "no backsliding." In other words, all existing programs will stay in place such that a trend towards attainment of air quality standards is maintained. Two exceptions to this approach are in the area of attainment demonstrations for existing standards and re-classification of existing ozone nonattainment areas.

### **How Political Lobbies Could Affect the Proposed Rule**

As demonstrated during the November 1996 elections, environmental issues have become extremely important from a political standpoint and the political debate over the need to revise the NAAQS for particulate matter and ozone has already begun. Leading the Republican charge is Senator John Chafee (R-RI), Chairman of the Senate Environmental & Public Works Committee. Chafee, who has historically stood strongly against conservatives in the 104th Congress seeking far-reaching environmental reform, drafted a letter to Administrator Browner demanding that U.S. EPA reconsider several key sections of the proposed standards. Specifically, the letter questioned the relative



uncertainty of the health effects science used by U.S. EPA to justify the revisions and the extent to which the public will benefit from the revisions. Although Chafee has taken a moderate stance on past environmental legislation, no Democratic signatures were received at the time the letter was submitted to U.S. EPA.<sup>21</sup>

In addition, Republicans are warning U.S. EPA that the proposed standards violate the 1980 Regulatory Flexibility Act, and are therefore likely to precipitate litigation from small businesses as well as intense congressional oversight early in the 105th Congress.<sup>22</sup> The primary issue is the apparent failure of the U.S. EPA to comply with a recent amendment to the Regulatory Flexibility Act known as the Small Business Regulatory Enforcement Fairness Act. President Bill Clinton signed the Act in early 1996 to ensure that federal agencies crafting rulemakings take into account the economic impact their rules will have on the small business sector. Under the amendment, all major rules are subject to review by an agency-convened small business advocacy review panel prior to proposal. However, U.S. EPA claims that the actual standard-setting proposals themselves will not have an economic impact on small entities, but will rather affect upcoming rules that specifically dictate how the revisions will be implemented.

In response to the hard-line position taken by Republicans, the Democrats have drafted their own letter, expected to be released to Administrator Browner in mid-February 1997. According to Republicans that have reviewed drafts of the letter, the letter serves only to provide a forum for U.S. EPA to defend their proposals. As the promulgation date nears, the debate over the economic burden compared to the health benefits realized is expected to become more intense from both sides of the aisle.

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<sup>21</sup> U.S. EPA's Clean Air Report, Vol. VIII, No. 2, p. 15, January 23, 1997. <sup>21</sup> U.S. EPA's Clean Air Report, Vol. VII, No. 25, p. 9, December 12, 1996.

<sup>22</sup> U.S. EPA's Clean Air Report, Vol. VII, No. 25, p. 9, December 12, 1996.

## Conclusions

The entire nation, including the federal government, states, and industry, have invested major efforts and resources to meet the current air quality standards for particulate matter and ozone. Despite this progress, U.S. EPA is proposing to change these standards based on what industry claims is limited creditable evidence. As a result of the proposed changes, industry and state regulatory agencies cannot ensure that the numerous control strategies developed to meet current standards will achieve compliance with the proposed NAAQS.

If promulgated, the proposed NAAQS for particulate matter and ozone could significantly affect the cottonseed oil mill industry. According to U.S. EPA and industry projections, the number of nonattainment areas resulting from the proposed NAAQS will triple, requiring facilities to deal with the complexities of federal nonattainment new source review permitting. The increase in nonattainment areas will affect approximately one-third of the country's cottonseed oil mills. The affected facilities are relatively large and account for over 40 percent of the nation's cottonseed processed.

At this time very little fine particulate matter data has been collected in the United States. Estimates have been made by U.S. EPA and others as to how many areas will initially be classified as nonattainment with the new standards, but without adequate data the full scope of the impacts of these new standards cannot be determined. Also, there has not yet been a U.S. EPA-approved method developed to sample wet emissions stacks. Lacking this method, many sources have been forced to assume that all emitted particulate matter from such a source is  $PM_{10}$ .

Although the short comment period does not allow industry to clearly and realistically determine the impact of the proposed changes, in general, industry strongly believes that the standards should be set at a level that is attainable under reasonable costs. Based on a review of the recent publications, industry feels that U.S. EPA has not been able to conclude that the proposed standards are justifiable on a health effects basis or are attainable under reasonable control strategies.

Superintendents should remain active in the rulemaking and regulatory development process of the proposed standards. Written comments regarding the proposed standards must be received by U.S. EPA no later than March 12, 1997. If possible, facilities should anticipate the possibility of future production increases and facility expansions that will require new source review permitting. It will be advantageous to permit these modifications prior to the implementation of the proposed new standards. Although intense debate between industry and U.S. EPA is expected until promulgation in July 1997, the cottonseed oil mill industry must be aware of these issues and the potential implications on future operations.





*New Technology of Plant Automation*

**Ronnie Sieber**  
Lubbock Electric  
Lubbock, TX

# 46th Oilseed Conference

**Processing Efficiency:  
Meeting the Challenge**

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Hotel Monteleone  
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Co-Sponsored by:

The American Oil Chemists' Society (AOCS)

The National Cottonseed  
Products Association, Inc.

Southern Regional Research  
Center/ARS/USDA





# **New Technology of Plant Automation**

**Ronnie Seiber**

**Technical Sales Rep.**

**for**

***Lubbock Electric Co.***



# ***Introduction***

- *Lubbock Electric Company, Inc.*
- *Automation Technology (New Controls)*



# *Lubbock Electric Co.*

Serving Industry Since 1944

# ***Diversified yet Focused***

## ***Departments Include:***

- **General Sales**
- **Motor Repair**
- **Air Compressors**
- **Hydraulics**
- **Control Systems**

# ***General Sales***

- **Modicon PLC's**
- **Square D PLC's, VFD's, MCC's**
- **Honeywell, Fisher, and Cashco Control Valves**
- **Honeywell, Fisher, and Kobold Process Instrumentation**
- **Total Control Products TouchScreens, Taylor MMI**
- **FactoryLink, Wonderware MMI**
- **U.S., Baldor, Magnetek Electric motors**
- **Quincy, Compair Kellogg air compressors**
- **Browning power transmission**
- **Sutorbilt blowers**
- **Sumitomo gearmotor**



# ***General Supply***

- *Bussman Industrial & Electronic Fuses*
- *Dayco V-Belts & Sheaves*
- *Simpson, Amprobe, Sperry Clamp-on Meters*
- *Browning Sprockets, Chain, Speed Reducers*
- *Aro Air Filters, Regulators, Lubricators*
- *Pioneer Refrigerated & Desiccant Dryers*
- *Milwaukee Power Tools*
- *Wiegmann, Rittal Electrical Enclosures*
- *Uninterruptible Power Supplies*
- *Misc. General Electrical & Industrial Supplies*



# ***Motor Repair***



- Rewinding of three phase, single phase, DC motors through 2500 HP
- VPI varnish available
- UL labelling
- Redesign HP, RPM, voltage
- Core testing, surge testing, balancing, machine shop services



# ***Air Compressors***



- Complete repair of reciprocal and rotary screw compressors
- Complete remanufacturing of positive displacement blowers
- Custom building of special compressed air systems
- Refrigerated dryers, filters, regulators, and accessories sold and serviced



# Hydraulics



- We handle Hugglunds Dennison hydraulic pumps
- Pumping unit design and manufacturing
- Vane and piston pump rebuilding
- Troubleshooting and remanufacturing



# *Control Systems*

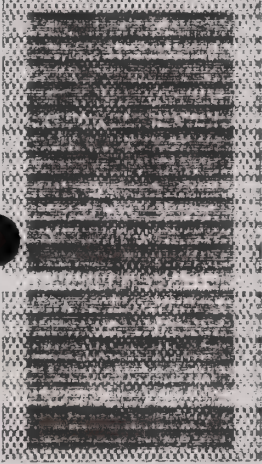
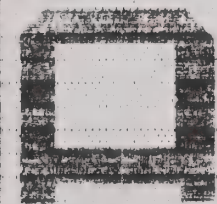


- Complete PLC and MMI system design and integration
- Automation, process control, and SCADA systems
- FactoryLink and Wonderware operator interface
- Total Control Products Operator Interface
- Panel and console fabrication
- Startup services



**AM** Magnetek

**Hamamatsu**



**WESTINGHOUSE**

**Ward**

**WIDUCHOW**

**Arbuck Electric Co**



(getting by with a little help from our friends)

**SUMITOMO**

**Quincy**

**Quincy Division**

**Quincy Industries**

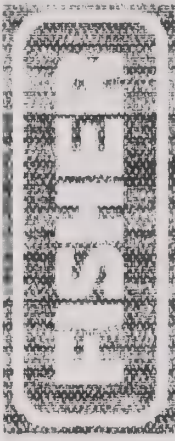


**ROSEMOUNT**

**SUTORBLT**

**ST/7/E**

**BEALDOER**



# *Topics of Discussion*

## *Five Main Things To Consider*

- *Process Control Needs*
- *What Controls Do I Need*
- *Expandability of New Controls*
- *How Do I Control My Process*
- *Personnel For New Controls*



# ***Process Control Needs***

- ***Monitoring Process***
- ***Partial Automation of Process***
- ***Total Automation of Process***
- ***Help on Making Decision***

# ***What Controls Do I Need***

- Distributed Control Systems
- Programmable Logic Controllers
- Single Loop Controllers
- Transmitter (Flow, Pressure, Temperature, and Level)
- Final Control Devices (Valves, Solenoids, Lights, and Alarms)



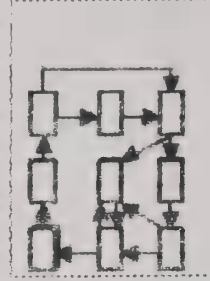
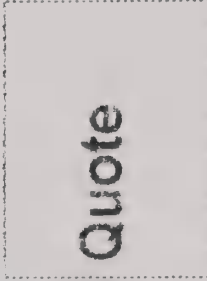
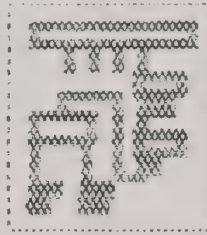
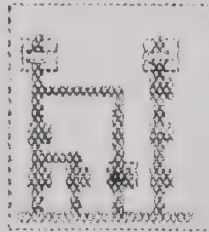
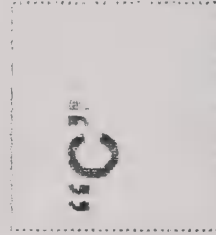
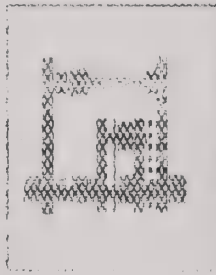
# ***Expandability of New Controls***

- Ability to add Extra I/O
- Ability to add new products (printer, Touch Screen, Operator Interfaces, Remote I/O Drops, Networking, Etc.)

*Use existing Tools or migrate to an even more productive environment*

Current

Ladder





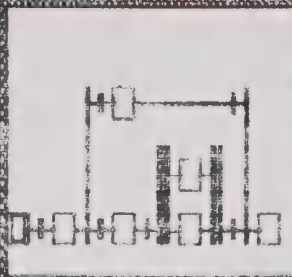
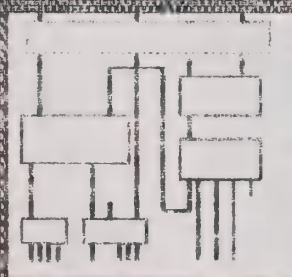
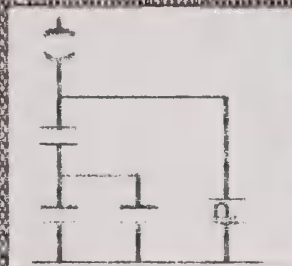
# Enhanced Language Set

## IEC LANGUAGES

Instruction Set, Symbolized and Ladder Diagrams

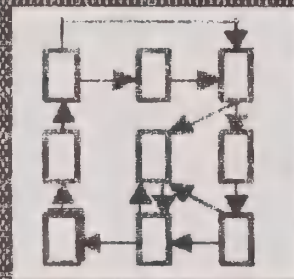
```
LD 15
ST C10.P1
LD %X10
ST C10.CU
CAL C10
```

```
J=104;
FOR I=1 TO 200 DO
IF W(I)=KEY THEN
J=1;
EXIT;
END IF;
END_FOR;
```

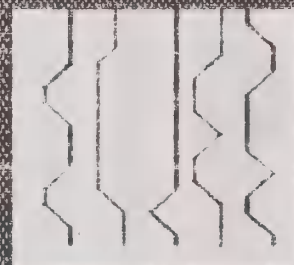


## Non IEC Languages

State Language Timing Diagram



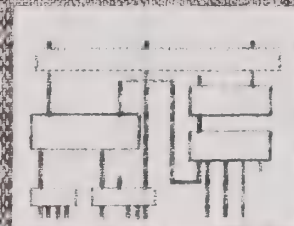
Standard Language



Motion Language

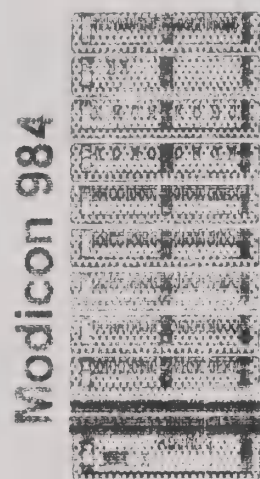
```
G1 Z 16.6 M73
G41 Y 1.25
G0
G1
X329 Y1.35
X128.86 Y 1.35
X300
G40 G41 R5
G0 Z 13
```

GAZ Language

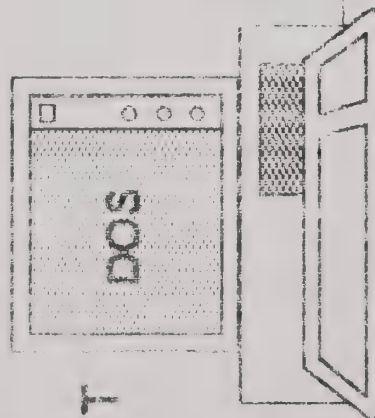




# Modicon Application Evolution



Modicon 984



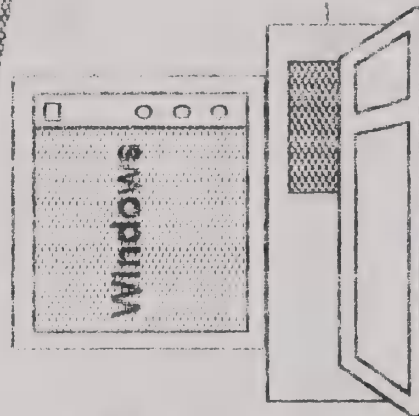
MODSOFT



## PORTABILITY

Translates program & configuration onto new platform

PROFIBUS  
WorldFIP  
ETHERNET  
MODBUS  
MODBUS +  
ASI  
Seriplex

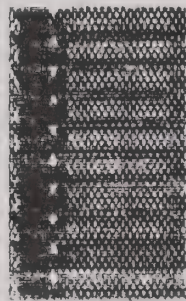


ConCept



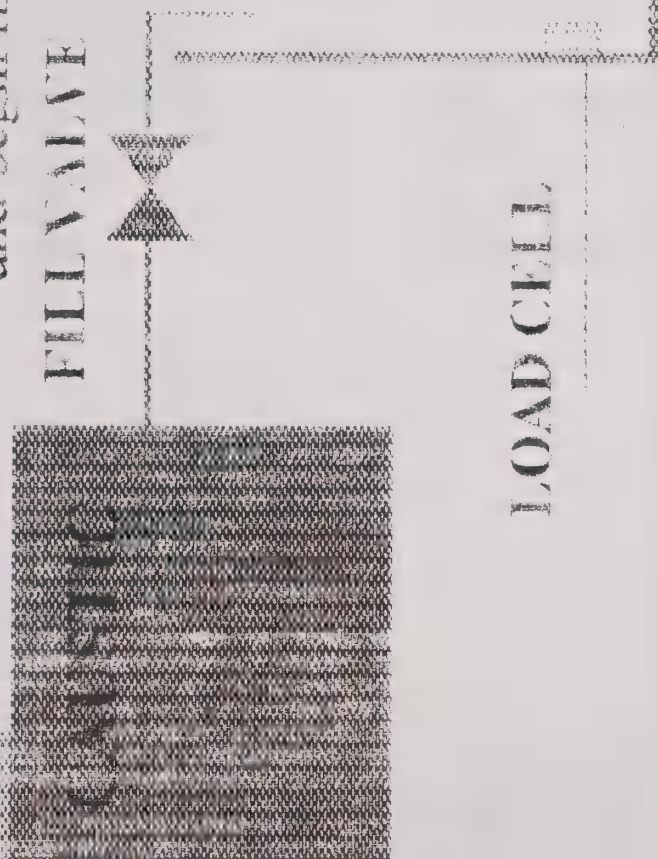
IEC 1131

Modicon Quantum



# MIX TANK EXAMPLE

When tank is full (load cell = 250) stop filling  
and run the agitator for three seconds.  
When agitator is done empty the tank (load cell = 0)  
and begin filling again.



Feel free to get creative with this problem. For example, add chemicals and pH probe. Agitate/empty based on density or turbidity instead of time.



# Lube Example

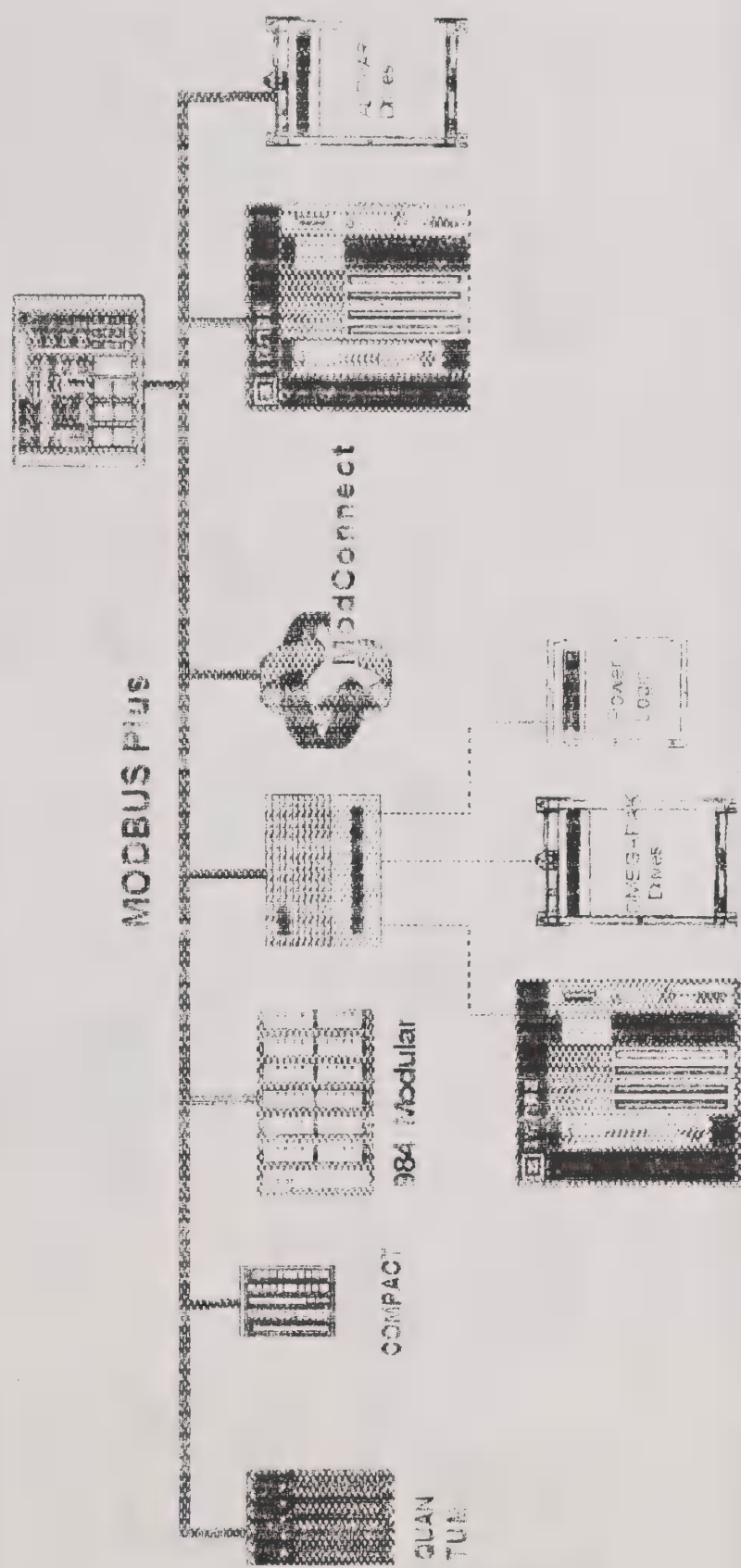


Timed: Run 30 sec, Lube 5 sec

Event: Lube for 2 strokes after each 20 strokes



# Expanded Options With MODBUS Plus

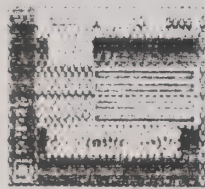


SYMAX

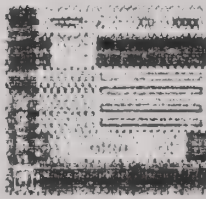
SYMAX  
Serial Interfaces

# Quantum Remote I/O Upgrade For SYMAX

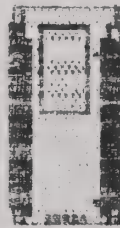
SYMAX



IONET Based  
(Twisted Pair)



SYMAX  
Register  
Rack I/O



Twisted Pair

Fiber Optics

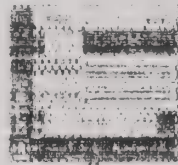
Fastest Throughput!

Longer Distances!

Protect Investment!

Quantum Remote I/O (Coax)  
or FIBER OPTICS

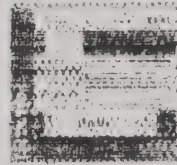
Quantum



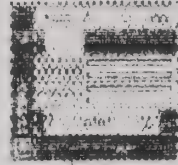
Quantum  
Register  
Rack I/O



Quantum  
Register  
Rack I/O



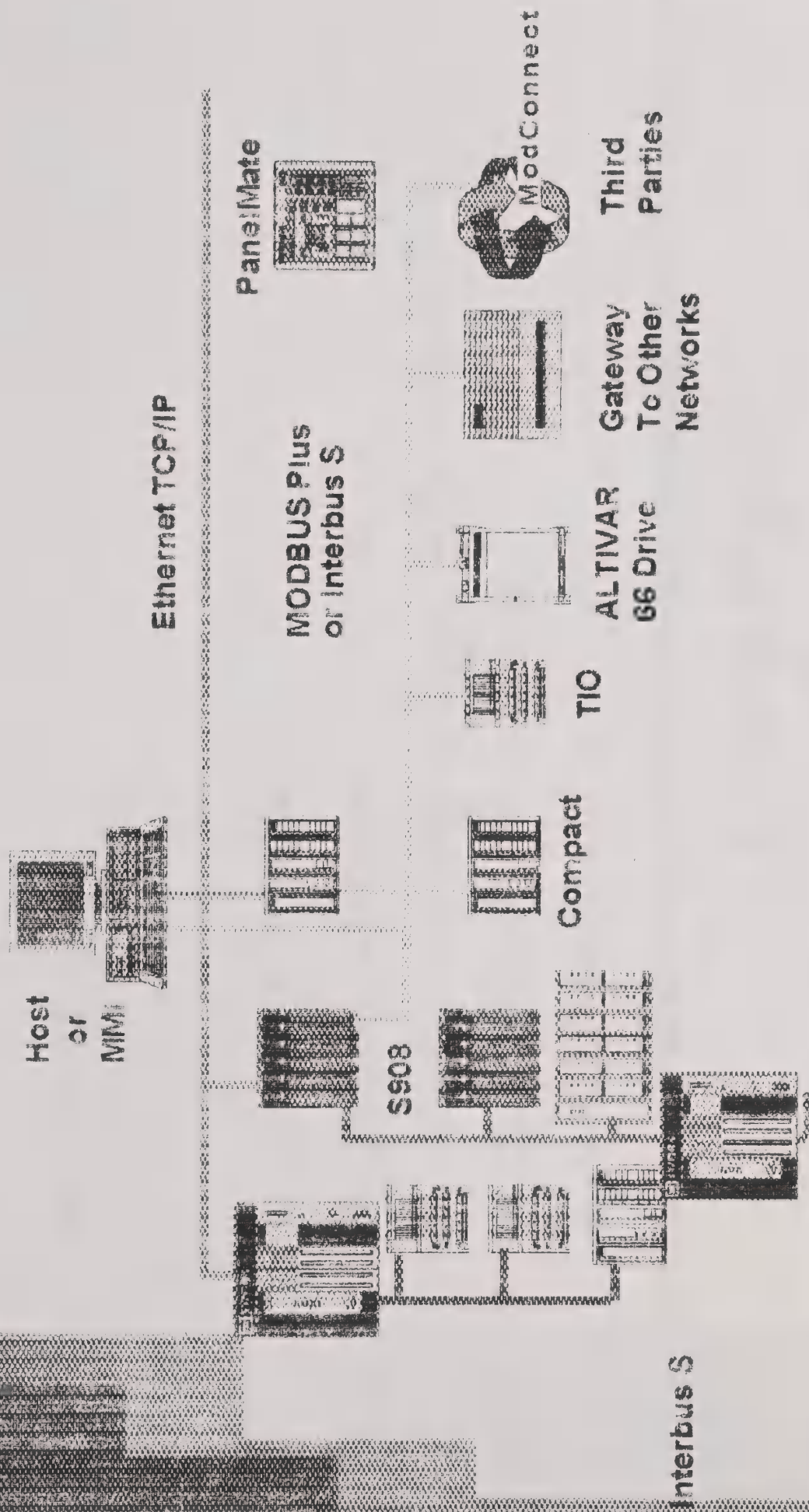
Quantum  
Register  
Rack I/O



Quantum  
Register  
Rack I/O



# Future Architecture





# ***How Should the Process be Controlled***

- **Engineering Firms**
- **Engineering Consultants**
- **Equipment Manufacture Engineering**
- **System Integration Houses**
- **Plant Personnel**

# ***Personnel For New Controls***

■ Operators

■ Maintenance

# Summary

- Summarize the five concerns for Automations
  - Process Control Needs
  - What Controls Do I Need
  - Expandability of New Controls
  - How Do I Control My Process
  - Personnel for New Controls



*Today's Research...Tomorrow's Impact*

**John Patrick Jordan**  
USDA, ARS, Southern Regional  
Research Center  
New Orleans, LA

# 46th Oilseed Conference

**Processing Efficiency:  
Meeting the Challenge**

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Hotel Monteleone

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Products Association, Inc.

Southern Regional Research  
Center/ARS/USDA



John P. ...

John P. ...

USDA, ARS, ...

...

...

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TODAY'S RESEARCH...TOMORROW'S IMPACT

JOHN PATRICK JORDAN

USDA, ARS, Southern Regional Research Center  
New Orleans, Louisiana 70179



The Agricultural Adjustment Act of 1938 gave birth to four regional research centers in the Agricultural Research Service (ARS), the purpose of which was to focus on utilization of agriculturally produced products by expanding current uses or inventing new uses. The focus will be in two directions: First, it will focus on the many significant research results that have emanated from the Southern Regional Research Center over its history, some in oilseeds and the remainder is from other areas of research. Secondly, I would like to discuss priorities in terms of ARS's program in oilseeds as the future unfolds.

Research accomplishments: Some of the efforts at the Southern Regional Research Center (SRRRC) have been focused on the inactivation, removal, and utilization of gossypol from cottonseed. The problem of gossypol in cottonseed is not new, but research efforts in this direction have improved the situation. In the late 1950's at the University of California, Davis, there was beginning to be substantial amount of research in the area of gossypol relative to cottonseed meal as a poultry

feedstuff. The possibility of solving this issue now looms on the horizon and the approaches would be to preferentially extract it or to produce cottonseed devoid of gossypol without sacrificing yield.

Another major issue relates to aflatoxin. You may be aware that Dr. Peter Cotty and his research group at the Southern Regional Research Center have put out large scale research plots in Arizona in cotton fields to see if in fact under field conditions *Aspergillus flavus* that does not produce aflatoxin can displace and beat out in the balance of ecology the *Aspergillus flavus* that does produce aflatoxin. Additionally, SRRC scientists have studied and now understand where and how the on-off switch functions relative to the production of aflatoxin. We are now advancing in a genetic approach to provide a plant that is much more resistant to aflatoxin infection in the first place.

These several approaches are being addressed simultaneously at the Center. Arizona was picked as the site to test non-aflatoxin producing *Aspergillus flavus* because of the dependability of aflatoxin infection on an annual basis and that an approach of displacing aflatoxin producers with non-producers

would be most desirable in that environment. Also, the non-aflatoxin producing strain is native to the area.

Most of you know that a large part of the research at SRRC relates to cotton. There is heavy emphasis on durable press, one of the outstanding inventions that is derived from putting chemical finishes on cotton which markedly reduces the need for ironing. One of the major problems, not for you and me who wear cotton, but for the workers in the plants that are putting on the durable press finish, is the exposure to formaldehyde. More recently, researchers at SRRC have found other ways of applying the durable press process completely devoid of formaldehyde, that uses polycitrates as garment finishing chemicals.

We have also had a continued interest in colored cottons, not because they are so popular, but because there is a question of a niche market and whether colored cottons which are by nature weak can be used in fabrics. Research at SRRC has developed a procedure whereby a center of stronger fiber, for example, normal white cotton, or even synthetic fibers can be used and colored cotton wrapped around it; the natural white cotton is a preferred core fiber.



The research on ascertaining and improving the strengths of cottons has been enormously successful. Today, using equipment, the development of which was contributed to by scientists at SRRC along with other ARS and industrial scientists, the industry has now been provided with a substantial amount of information about the quality of the cotton while still in its raw state. This has led to the United States of America enjoying a premium price for cotton around the world and has led to the use of this equipment on a regular basis by USDA's Agricultural Marketing Service in evaluating the quality of cotton.

Two items of cotton research at SRRC which are of interest to cotton weavers are the techniques in the chemistry associated with dyeing and finishing, an area that has made major strides in recent time. The brightness and durability of the dyes and finishing that are used today on cotton products are enormously improved because of our research efforts compared with that of years past. Moreover, the importance of the discovery of dyeing and finishing chemicals that are non-irritant and non-allergens is an important research advancement.

A related area is the modeling of cellulose, which involves very basic studies to understand the nature of the molecules

involved in retaining dyes. The studies show what can and cannot fit in and around those molecules and what chemical reactions are most likely to be effective. This is one of the important reasons why dyeing and finishing today is substantially more successful than it was in earlier decades.

Moreover, the possibility of using cellulose as a starting substrate for the development of new products is extremely promising, such as adhesives, coatings, and plastic-like materials that lend themselves to biodegradability when placed in a land-fill situation. The scientific staff at SRRC has recently had a new addition in the form of a collaborator from the Hawaii Agricultural Research Center who is studying, with us and in collaboration with other scientists at Southern Mississippi University and elsewhere in the nation, and these new products from sucrose are being developed. The visiting scientist, Dr. Nozar Sachinvala, is interested in moving beyond sucrose into cellulose with the same kind of questions and possibilities of products. A new use for cottonseed may be this area of exploration.

There are, however, a number of other research areas at the Center, including flavor research, research on the nature food

components and their use in food products and as additives to food products. Among the areas that are also being studied are techniques to keep fresh cut fruits and vegetables fresh longer, and to lengthen their shelf life quality.

Tied in with this is sensory evaluation which involves the training of panelists over an extended period of time (literally months) so that they can rate the intensity of flavor components. This differs dramatically from another type of food evaluation which simply asks whether this product tastes better to you than another similar product. What we are hunting for here is the determination of the presence of specific compounds that influence flavor, palatability, aroma, etc., of food. Hitched to that is an extensive research program to study through analytical instrumentation the chemical structures of the compounds that are identified as flavorful or undesirable; therefore, providing industry with a capacity to use instrumentation to detect the presence or absence of plus factors and minus factors in food flavor.

SRRC scientists are also studying formulations encapsulated in a pasta like product which allows the slow release of biocontrol agents. The formulations keep the biocontrol agents



alive and are placed at the base of the plant. The pasta is then dissolvable over time in water when the ground is irrigated or it rains. The pasta acts as a nutrient source and home for the biocontrol agent allowing its timely release. Perhaps the technique could even be used with fertilizer.

SRRC is deeply involved in sugar research, not just in terms of new products from sucrose but also the development of varieties suited for specific environments. SRRC is also interested in the processing of sugar, and this research is being conducted at SRRC by our own scientists and those of the Sugar Processing Research Institute and more recently the Hawaii Agriculture Research Center, both collaborating research groups at SRRC.

I have just scratched the surface in summarizing some of the things that have happened at the Center over a period of half a century or more. Many research accomplishments are recorded by scientists at the Center, having been given over 1,000 patents on agriculturally related products and processes and having published over 9,000 scientific research papers. In the 1970's, a study was published addressing the productivity of the utilization laboratories in the first 25 years of life. The

Jones study was very interesting in that it provided evidence that it only took two discoveries, just two discoveries to pay for all of the facilities, all of the staff, all of the equipment, and all of the operating money for the four utilization centers during that entire quarter century. One discovery was durable press cotton which was developed at SRRC and has been in use not only in the US but around the world. And the second was the development at the Peoria laboratory of a method to rapidly produce penicillin which made a very inexpensive readily available antibiotic. A similar study to look at the second quarter century of the utilization centers has been initiated.

Let us now turn our attention to priority setting in ARS and in science in general. Both ARS and the universities depend significantly on input from industry, consumers and users. As you make that input, it is good for you also to recognize not only what you need, but tell us what we have done right. At the same time, tell us what we have done that is not so right, that is to say, new technologies that have left you in a more vulnerable or perhaps economically disadvantaged position in terms of international trade. All systems, particularly ARS's system, depend on input from industry, consumers and users.

The second stage is for scientists (scientists within ARS, and those who cross the scientific boundaries or organizational boundaries which would include universities) to provide input as well as digest the input from industry, consumers and users. Input is needed to determine what is "doable," what is economically impactful, where is the pressure and importance of doing this aspect of research vis-a-vis another one. This provide a second level of input into the decision making process.

For us within ARS, the National Program Staff (NPS) is a major player in decisions about research. These are National Program Leaders who are particularly selected because of their breadth of knowledge of the problem area and of the research emphases of scientists, both within and outside of the ARS. Finally a decision is made, often in dialogue between the research scientist and the NPS leadership together with the Center Director, to ascertain priority research.

There is yet another level of input, and that is the Congress of the United States. They often put specific instructions into the Appropriations or Authorizing Acts that define critical program areas.



Current priority research areas: Tung oil is a good drying oil because of the high concentration of eleostearic acid. Tung oil production which has been dormant in the United States since 1968, has recently been started again in Mississippi. This is a rejuvenation of domestic production which at one time was really quite high. In 1958, the United States produced 44.8 million pounds of tung oil. Today we import most of it. Scientists at SRRC have been examining Chinese melon seed as a source of drying oil since it contains 44% oil with eleostearic acid accounting for 65 percent of the fatty acids in. The potential for Chinese melon seed oil is high since its triglyceride composition is similar to tung oil. SRRC scientists are researching the potential for extracts to convert linoleic acid to eleostearic acid, thus increasing the potential for the Chinese melon to be an excellent source for drying oil.

SRRC scientists also have learned that ethanol containing small amounts of citric or phosphoric acids can liberate chemically bound gossypol permitting its removal from cottonseed meal, thus making it a more desirable feed stuff. Scientists are now addressing the issue of consistency of removal of free gossypol and other scientists have addressed the genetics of the cotton plant to determine if a seed can be gossypol free or close

to it. This work has been going on for three or four years and the two major approaches are breeding versus extraction. Obviously these are not mutually exclusive and headway is being made.

Although gossypol is not a new problem, we have learned a great deal about managing the presence of gossypol in feed stuffs mainly through research done in feed formulations where addition of divalent iron binds gossypol. Collaborative research has been conducted using chickens, lambs and pigs as experimental animals. Since the overall plant contains about 1% gossypol upon extraction, this co-product needs a "home" or a use. It is an excellent antioxidant but very powerful. It also has anti-virus and anti-tumor characteristics. This of course was reported to you in some detail by a scientist from the University of New Mexico about three years ago. In any case in terms of priorities for SRRC and ARS, gossypol is still among the top priority issues. Even though work has continued on this problem for half a century, progress is slowly being made.

Further, extraction technologies and improvement therein remains a high priority for ARS. This is true not only with respect to the removal of gossypol using acetone or acetates or

other polar solvents, but also the possible involvement of phospholipids in the process. Clearly there are some problems associated with this approach. Overall we have learned that quality and increased efficiency in extracting oil can be had by adding specific lipid components and then using isohexane as the extractant.

One of the brighter potentials is to use the double extruder as a way to improve the productivity and make it easier to manage an extraction system. It will have the benefit of significantly reducing free gossypol in the cottonseed meal. Currently research focuses on the use of expanders to improve the rate of removal of oil and to produce consistently low free gossypol meal.

Having laid out a "draft scenario" of oilseed research, we invite your comments. It is through such dialogue that ARS and especially SRRC can remain "on the mark" in terms of the priority of its research efforts.





*Update on Methods to Prevent Aflatoxin  
Formation*

**Peter J. Cotty**

USDA, ARS, Southern Regional  
Research Center  
New Orleans, LA

# 46th Oilseed Conference

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Southern Regional Research  
Center/ARS/USDA







### **Update on Methods to Prevent Aflatoxin Formation**

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#### **Summary**

Aflatoxins are cancer causing chemicals that may form in cottonseed both during boll development and after bolls have opened. These toxins are produced by certain strains of the fungus *Aspergillus flavus* during infection and growth in cottonseed. The environment, insect damage, moduling, and harvest and gin dates all greatly influence the quantity of aflatoxin in the cottonseed at market. Aflatoxin content can increase in seed until use. Both traditional management practices and new technologies may be useful in limiting contamination. Timely harvest and ginning can greatly reduce the severity of contamination, and proper sorting of the seed at ginning and at receipt can greatly reduce the incidence of contaminated lots and increase the reliability of analyses. Timing of irrigation, fertilization, and insect control all influence contamination. Bt cotton can limit insect damage to developing bolls and thus reduce contamination. However, exposure of mature seed to environments favorable to contamination may cause Bt cottonseed to also become contaminated with unacceptable aflatoxin levels.

The use of atoxigenic strains (strains which do not produce aflatoxins) of *Aspergillus flavus* to prevent contamination is another new technology. Atoxigenic strains of *A. flavus* reduce contamination by competitively excluding aflatoxin producing strains.

The use of atoxigenic strains has the potential to reduce contamination both during boll development and after boll opening. In 1996, the U. S. Environmental Protection Agency granted the U. S. D. A. permission to treat a limited commercial cotton acreage with atoxigenic strains of *A. flavus* in order to assess efficacy in and compatibility with commercial agriculture. These tests will also help define the potential of atoxigenic strain applications to provide long term and area wide reductions in crop vulnerability to aflatoxin contamination.

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Aflatoxins are toxic, carcinogenic chemicals that frequently occur in foods and feeds. Health concerns have led to regulatory limitations on the aflatoxin content of foods throughout most of the world (Stoloff, et al. 1991). The most toxic and highly regulated aflatoxin is B<sub>1</sub> (Park and Stoloff, 1989; Stoloff, et al. 1991). The fungus, *Aspergillus flavus*, causes aflatoxin contamination of cottonseed. Contamination results in losses for producers, processors and animal industries that depend on cottonseed for feed (Park and Stoloff, 1989). Whole cottonseed and/or cottonseed products are an important dairy and cattle feed. Aflatoxins in cottonseed are transferred to milk in slightly modified form (Park, et al., 1988; Robens and Richard, 1992). U.S. regulations prohibit aflatoxin concentrations over 0.5 µg/kg in milk. Milk may be destroyed and entire operations temporarily shutdown and quarantined in dairies producing milk tainted with unacceptable aflatoxin levels (Emnett, 1989). To prevent unacceptable

aflatoxin levels in milk, the regulatory threshold for aflatoxin B<sub>1</sub> in cottonseed fed to dairy cows is 20 µg/kg (Park, et al., 1988). Cottonseed containing less than 300 ppb aflatoxin B<sub>1</sub> can be fed to mature beef cattle. Greater aflatoxin quantities prevent cottonseed use as a feed. Failure of cottonseed to meet dairy requirements results in a decrease in cottonseed value; aflatoxin content is often the most important factor determining the value of whole cottonseed.

All cotton producing regions of the U.S. may experience some aflatoxin contamination in some years. However, in most of the cotton belt occurrence of aflatoxin levels unacceptable for dairy use is infrequent (Russell, 1980). In other regions aflatoxins are a perennial concern. These regions include the production areas of Arizona, southern Texas, and the Imperial Valley of California.

Aflatoxins contaminate cottonseed when *A. flavus* infects the seed either during boll development or after boll maturity (Cotty, 1991). During boll development, contamination may occur when *A. flavus* infects through wounds or cracks. In Arizona, pink bollworm exit holes are the most common factor that has been implicated in predisposing bolls to infection during this first contamination phase. Bolls infected during this first phase typically produce seed with fluorescent staining (bright-green-yellow-fluorescence = BGYF) on the lint and linters. Although seed with linter BGYF typically composes less than 1% of the crop, individual BGYF seeds may contain aflatoxin in excess 500,000 PPB (Lee, et al., 1990). During some years, seed infected during this first phase may contain most of the aflatoxin. The second phase of contamination occurs when mature seed is exposed to both conducive temperature [usually above 30 C (80 F)] and either high relative humidity (above 85%) or rewetting



at or after boll opening. This phase is characterized by increases in aflatoxin content of seeds infected during the first phase, as well as, infection of new seed (Cotty, 1991).

The second phase of contamination can occur in the field, in modules, and even in seed piles at dairies. In areas where aflatoxin contamination is a perennial problem, seed cotton becomes associated with *A. flavus* propagules shortly after boll opening and seed infection may proceed when the seed is exposed to adequate moisture. Seed infected during the second phase often do not exhibit BGYF on either linters or lint.

### **Limiting Contamination with Traditional Methods**

Aflatoxin contamination of cottonseed can be limited by careful agronomic practice. Insect control measures must be used to reduce damage to developing bolls, particularly when the crop is being produced under hot dry conditions. Insect damaged bolls may contain the most aflatoxins in certain years. Much of the contaminated seed from these bolls typically exhibits bright-green-yellow fluorescence (BGYF) on the linters. However, in some years most of the contamination is formed after bolls open when the crop is exposed to high humidity (late irrigation, dew, rain, fog) either in the field, in modules or in storage. This second phase of contamination may be limited by harvesting the crop early, properly constructing modules with dry seed cotton, tarping modules, and protecting the ginned seed from high humidity until use. This includes during storage at dairies. The crop should be managed to limit vegetative growth and reduce canopy density. Reducing the frequency of irrigation and fertilization may facilitate this. Late irrigations are known to increase aflatoxin contamination (Russell, *et al.*, 1976). Unfortunately, even under careful management, unacceptable aflatoxin

levels may occur via either unpreventable insect damage to the developing crop (Cotty and Lee, 1989), or exposure of the mature crop to moisture prior to harvest (Cotty, 1991a), during storage in modules (Russell and Lee, 1985), handling, transportation, or even use (Cotty, 1991b).

Seed from first-pick cotton should always be segregated from ground-gleaned cotton at the gin. Although practical for relatively few gins, keeping seed from different fields distinct until after aflatoxin content is known can increase the number of seedlots with acceptable aflatoxin. Proper sorting upon receipt of seed at mills can also limit losses attributable to aflatoxins.

### **Transgenic Bt Cottonseed Can Help Limit Aflatoxin Contamination**

Insect management is an important component of traditional management of aflatoxin contamination and because transgenic Bt cottonseed is resistant to pink bollworm damage, it holds the promise of being an important component of sound aflatoxin management programs. Pink bollworm exit holes are important avenues through which aflatoxin producing fungi infect developing cottonbolls (Ashworth, *et al.*, 1971; Russell, *et al.*, 1976; Cotty and Lee, 1989). Therefore, prevention of pink bollworm damage to the developing crop is an important component of programs directed at managing aflatoxin in areas where the environment predisposes the crop to contamination. Cultivars resistant to pink bollworm, such as certain transgenic Bt cottons, may facilitate pink bollworm management. However, pink bollworm damage is not directly correlated with aflatoxin contamination (Henneberry, *et al.*, 1978; Russell, *et al.*, 1976) and unacceptable aflatoxin concentrations may contaminate seed

from fields with no pink bollworm exit holes (Russell, 1980). Thus, other factors must also play decisive roles in determining the quantity of aflatoxin in the crop.

Although pink bollworm resistant cultivars can be expected to have less aflatoxin contamination than susceptible cultivars when pink bollworm pressure is high, these cultivars are not immune to aflatoxin contamination per se. Any physical damage may predispose developing bolls to aflatoxin contamination. Insects other than pink bollworm and certain types of physiological stress (*i.e.*, heat stress-induced suture cracking) may also predispose bolls. Furthermore, a second phase of infection occurs when mature seed (open bolls through ginned seed) is exposed to adequate humidity and temperature to permit aflatoxin producing fungi to grow and contaminate the seed. This second phase apparently led to unacceptable aflatoxin contamination of several commercial lots of Bt cottonseed in 1995 and 1996. This is supported by observations on one highly contaminated commercial seed lot (contained greater than 6,000 ppb aflatoxin B<sub>1</sub>) where most contamination occurred in seed without BGYP. Typically, some or all seed produced in locks infected during the first phase of contamination will exhibit some linter BGYP. As bolls fluff-out and dry, the capacity of lint and linters to support BGYP formation is lost. Thus, aflatoxin contaminated seed lacking BGYP may reflect infection subsequent to boll splitting. The commercial seed lot discussed above had a free fatty acid content (1.69%) that was elevated compared to the norm for the region. Free fatty acid increases are also associated with exposure of seed to high humidity (Conkerton, et al., 1989). See the Proceedings of the 1997 Beltwide Cotton Production Conference (Aflatoxin Contamination of Commercially Grown Transgenic



Bt Cottonseed, P. J. Cotty, D. R. Howell, C. Bock, and A. Tellez, in press) for details of our work on aflatoxin contamination of Bt cottonseed.

### **Atoxigenic Strains of *Aspergillus flavus* may be the Key to Reliable Management**

A new technology is being developed to prevent aflatoxin contamination. This technology causes the community of fungi resident in cotton fields to have a lower potential to produce aflatoxins. Fungal communities composed of *Aspergillus flavus* are highly complex and are composed of strains which differ morphologically, physiologically, and genetically (Bayman and Cotty, 1993; Cotty, 1989). Differences among strains in ability to produce aflatoxins is well known (Cotty, *et al.*, 1994) and aflatoxin producing ability is not correlated with strain ability to colonize and infect developing cotton bolls (Cotty, 1989b). These observations led to the suggestion that atoxigenic strains of *A. flavus* might be used to exclude toxigenic strains through competition during infection of developing crops and thereby prevent aflatoxin contamination (Cotty, 1989b; 1994). In both greenhouse and field experiments, wound inoculation of developing cotton bolls and corn ears simultaneously with toxigenic and atoxigenic strains led to reductions in aflatoxin contamination of the developing crop parts as compared with controls inoculated with only the toxigenic strains (Cotty, 1990; 1992). Atoxigenic strains are effective at preventing post-harvest aflatoxin contamination both when the crop is infected naturally in the field and when inoculated after harvest (Brown, *et al.*, 1991). Thus, competitive exclusion of aflatoxin producing strains of *A. flavus* with atoxigenic strains of the same fungal species may provide a

single method for preventing aflatoxin accumulation throughout crop production and utilization (Cotty, *et al.*, 1994).

In order for atoxigenic strains of *A. flavus* to be useful during crop production, they must be applied at a time and in a manner that allows them to compete successfully with aflatoxin-producing strains. In theory, application of an atoxigenic *A. flavus* strain early in the season should give the atoxigenic strain preferential exposure to the developing crop and thus the advantage in competing for crop resources during infection and during *A. flavus* community increases associated with cultivation (Cotty, *et al.*, 1994).

An aflatoxin prevention technology based on atoxigenic strains of *Aspergillus flavus* is being developed for use in the region of Arizona with the most frequent and severe aflatoxin contamination of cottonseed. Strains are seeded into cotton fields at lay-by (immediately prior to first bloom). The strains are applied to the soil surface under the crop canopy in the form of colonized sterile wheat seed. When the crop is subsequently irrigated, the atoxigenic strain utilizes the resources in the colonized wheat seed, sporulates, and disperses to the crop. Wheat seed colonized by atoxigenic strain *Aspergillus flavus* AF36 has been evaluated in small scale test plots since 1989. Strain seeding caused large and significant changes in the *Aspergillus flavus* community on the crop and in the soil. Applications resulted in the applied atoxigenic strain becoming dominant in the field and aflatoxin-producing strains becoming less frequent. These changes in the *A. flavus* communities were associated with great reductions (75% to 99%) in aflatoxin contamination (Cotty, 1994). Further tests showed that atoxigenic strain applications have a long term influence on *A. flavus* communities resident in agricultural fields. This suggests atoxigenic strain applications may have benefits over

multiple seasons and that long-term, area-wide changes in the aflatoxin-producing potential of *A. flavus* communities may be achieved. Results of field plot tests indicate that atoxigenic strain applications do not increase the amount of *A. flavus* on the crop at maturity and do not increase the percent of the cottonseed crop infected by *A. flavus*.

*Aspergillus flavus* typically becomes associated with crops in the field during crop development, and remains associated with the crop during harvest, storage and processing. Thus, crop vulnerability to aflatoxin contamination remains until the crop is ultimately used. Similarly, atoxigenic strains seeded into agricultural fields prior to crop development will remain associated with the crop until use and may provide long-term postharvest protection from contamination.

Development of atoxigenic strain based management is proceeding with performance of large scale commercial tests. These tests will determine how to fit the technology into commercial practice and how to assess benefits of large scale applications. Because atoxigenic strains are considered biopesticides, such evaluations require entry into the pesticide registration process and the granting of an Experimental Use Permit and an Exemption from Tolerance by the U.S. Environmental Protection Agency. With the help of the Interregional Research Project No. 4, the Agricultural Research Service has entered into the registration process and has obtained an Experimental Use Permit to treatment commercial acreage in 1996 through 1998. The National Cotton Council, National Cottonseed Products Association, and growers, ginners, and crushers all played an important role in the registration process by providing supporting comments to the EPA.



The experimental program outlined in the Experimental Use Permit calls for treatment of 120 acres in 1996 and 500 acres per year in 1997 and 1998. In collaboration with gins and growers in Yuma County, Arizona, an experimental plan was designed to comply with this program. The first treatments to commercial fields were initiated in June 1996. The product was applied (10 lb. per acre) to three commercial fields by growers at lay-by with widely available Gandy boxes. The product performed as designed and effectively delivered the atoxigenic strains to the crops. Crops produced in treated fields met or exceeded control field crops in yield and quality, and preliminary data indicates efficacy in reducing contamination of the commercial crop. Atoxigenic strains may become valuable tools for limiting aflatoxins in cottonseeds.

#### **Aflatoxin Prevention in a Nutshell**

The environment, insect damage, moduling, and harvest and gin dates all greatly influence the quantity of aflatoxin in the cottonseed at market. Aflatoxin content can increase in seed until use. Both traditional management practices and new technologies may be useful in limiting contamination. Timely harvest and ginning can greatly reduce severity of contamination, and proper sorting of the seed at ginning and at receipt can greatly reduce the incidence of contaminated lots and increase the reliability of analyses. Timing and frequency of irrigation, fertilization, and insect control all influence contamination. Bt cotton can limit insect damage to developing bolls and thus reduce contamination. However, exposure of mature seed to environments favorable to contamination may cause Bt cottonseed to also become contaminated with unacceptable

aflatoxin levels. If the insect protection provided by Bt cotton means growers will hold the crop in the field longer, the benefits of Bt cotton to management of aflatoxin contamination may be lost. The use of atoxigenic strains of *Aspergillus flavus* to prevent contamination is a new technology that should improve aflatoxin management under most conditions. Atoxigenic strains of *A. flavus* reduce contamination by competitively excluding aflatoxin producing strains. The use of atoxigenic strains has the potential to reduce contamination both during boll development and after boll opening. In regions where aflatoxin contamination is frequent and severe, aflatoxin contamination will be most reliably limited by combining new management technologies with traditional techniques.

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*New Technology Development  
at Texas A&M University*

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College Station, TX

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## **“New Technology Development at Texas A&M University”**

by

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The development of new technology for oilseed processing continues to be one of the primary activities at the Food Protein R&D Center (FPRDC) at Texas A&M University. Activities at this fifty-five year old institution include innovative research in the areas of extrusion, membrane separation, refining, and developing new products from existing raw materials. As a way of informing industry of this research, the FPRDC presents a series of short courses, publishes articles in numerous technical and scientific journals and participates in meetings and conferences.

These activities support the goals and objectives of the FPRDC. The Center works to bring benefits to Texas Agribusiness through:

- \* Process improvement
- \* New process/product development
- \* Add value to existing products
- \* Technical assistance
- \* Information dissemination
- \* Trouble shooting
- \* Short courses
- \* Agrochemical registration

The various activities at the Center are divided into six program areas:

- \* Basic Sciences Program
- \* Extrusion Technology Program
- \* Fats and Oils Program
- \* Separation Sciences Program
- \* Industry Services Program
- \* Good Laboratory Practices Program

Current research at the Center involves projects discussed below:

**Modification and Fractionation of Vegetable Oils by Directed  
Interesterification and Crystallization  
Dr. E. Hernandez and Dr. K. C. Rhee**

A continuing project to develop techniques for removal of saturates through interesterification and crystallization at a pilot plant scale; to develop a process to fractionate fats and oils without the use of refrigeration; to evaluate the physical and organoleptic properties of high-melting cottonseed oil stearin and olein; and to apply the technique to other fats such as tallow and other fats and oils of interest to Texas.

**Development of Biodegradable Material from  
Agricultural Crops Grown in Texas  
Dr. M. N. Riaz & Dr. K. C. Rhee**

A new project to generate fundamental understanding of the physical, mechanical and rheological behavior of various biodegradable plastics, which may be derived from soybean, cottonseed, rice, corn, and other agricultural crops produced in the State of Texas and to gain ideas on the development of competitively priced biodegradable packaging for food applications.

**Bioconversion of Soybean processing Waste, Soapstock and Bleaching  
Clays into Organic Fertilizers and Soil Conditioners  
Dr. S. S. Koseoglu, Dr. S. Wale & C. Vavra**

A new project to develop procedures to convert oilseed processing wastes, used bleaching clays and crude gums into organic fertilizers and soil conditioners to improve the profitability for oilseed processing industries by establishing new markets for their crops, create new jobs and reduce pollution and other environmental problems. These products have a wide range of applications, i.e., production of organically-grown foods and maintenance of golf courses and parks and care of home gardens and plants.

**Modification of Structure of Oilseed Proteins for Improved Properties  
Dr. K. C. Rhee, Dr. S. K. Lee & Dr. K. S. Kim**

A continuing project to systematically study basic mechanisms of modifying structures of protein molecules to produce ingredients with improved functionality, characterize them for chemical, physical, nutritional and functional properties, test them in new food, feed and industrial applications, and evaluate feasibilities of scaling up the new technologies into economically viable commercial processes.

**Recovery of Value-added By products from Cottonseed and Other  
Vegetable Oil Gums and Soapstock**

**Dr. E. Hernandez, Dr. S. S. Koseoglu & Dr. K. C. Rhee**

A new project to recover antioxidant compounds such as tocopherols, sterols, specific lecithin fractions and fatty acids from gums (phosphatide fraction) and soapstock by fractionation using solvents such as hexane, isopropanol, ethanol and acetone and molecular distillation and characterize the products for their antioxidant activities in different oils.

**Value added Poultry Feed from Texas Poultry Waste**  
**Dr. M. N. Riaz & Dr. K. C. Rhee**

A new project to evaluate the feasibility of processing poultry byproducts and eggshells into a value-added poultry feed by extrusion and other techniques.

**Polyurethane Foams from Cottonseed Oil Byproducts and Their Utilization in  
Construction Insulation Industries**  
**Dr. S. S. Koseoglu, Dr. S. Wale & C. Vavra**

A new project to convert triglycerides and phospholipids into polyols and produce a new generation of polyurethane foams from cottonseed oil, formulate and modify cottonseed oil-based polyurethane to obtain different physical properties, evaluate chemical and physical properties of these new products, and test them in selected areas of construction-insulation applications.

**Deactivation Allergens in Major Oilseed Meals and Proteins by Extrusion**  
**Dr. K. C. Rhee, Dr. K. S. Kim, Dr. K. H. Lee & Dr. M. N. Riaz**

A continuing project to develop techniques for allergenicity tests, processes for preparing allergen-free soybean meals and proteins for use in milk replacers, starter feeds for young animals and pet foods; to develop procedures for preparing allergen-free, low gossypol content cottonseed meals and proteins, and survey their potential applications in feeds of young ruminants, swine, poultry and aquaculture feeds.

**Environmentally Friendly Extruder Aquaculture Feeds**  
**Dr. M. N. Riaz & Dr. K. C. Rhee**

A new project to develop and document extrusion processing techniques for delaying disintegration and solubilization of extruded sinking and floating feeds for shrimps and catfish in various sizes used from larvae to growout stages, prepare prototype products for feeding trials to ensure that nutrients are retained and attractants are still released.

**New Simple, Effective and Energy Efficient Hexane Vapor Recovery Process**  
**Dr. S. S. Koseoglu, C. Vavra, S. R. Gregory**

A new project to replace the mineral oil absorption system with a simple, energy efficient and effective membrane-based hexane vapor recovery system. Process optimization and on site testing will be conducted at the Center's pilot plant.

**Purification and New Markets for Gossypol Products**  
**S. R. Gregory, K. S. Kim & K. C. Rhee**

A continuing project to separate and purify gossypol for use as drugs and a chemical feed stock. Two methods that do not depend on the soapstock as the raw material will be examined: extraction of gossypol from flakes or expanded collets with cold ethanol or isopropanol. This method will require several steps of purification and crystallization to remove sugars that are extracted with gossypol. The other method involves extraction of gossypol from the expander-processed cage oil which contains as much as 1.5% gossypol. Membrane separation of gossypol from the oil-hexane miscella will also be examined.

**Conversion of Cottonseed Oil to Transformer Oils**  
**Dr. S. S. Koseoglu & C. Vavra**

A new project to: device a practical and economic process to convert vegetable oils into methyl esters; determine operational conditions for esterification and partial hydrogenation; identify operational problems and their probable solutions; and determine economic feasibility of the process to increase economic benefits to cotton growers.

**Reduced Gossypol Content Meals for Targeted Markets**  
**S. Gregory, S. Doty, W. H. Johnson & Dr. K. C. Rhee**

A continuing project to identify specific markets, their needs and potential volume for reduced-gossypol content meals; develop and optimize techniques for reduction of free gossypol in cottonseed meals through binding by heat and moisture treatment and using feed additives such as iron salts or extract gossypol with various solvents, to decrease gossypol toxicity for feeding to non-ruminants and possibly for additional feeding to ruminants.

**Small-scale Processing of Oilseeds and Cereals**  
**M. F. Gerngross, Dr. K. C. Rhee**

A continuing program that provides small-scale processing services of oilseeds and cereals for agrochemical industries on a self-funding basis.



**Application of Silicate Refining Agents in Cottonseed Oil Processing:  
Elimination of Water Waste and Improvement of Oil Quality  
Dr. K. C. Rhee & Staff**

A new optional project to determine the optimum conditions for using soluble silicates to eliminate free fatty acids, and remove gums, gossypol and color from the crude oil; to evaluate the use of causticized insoluble silicates to remove free fatty acids by filtration; and to evaluate the final RBD oils for free fatty acids, peroxide, p-anisidine, metal, gossypol, antioxidant changes and flavor.

If any of the above projects are of special interest to you and you would like more information, please contact the Food Protein R&D Center at Texas A&M University.



*Whole Cottonseed Research & Promotion  
Program at Cotton Incorporated*

**T.C. Wedegaertner**

**W.F. Lalor**

**Cotton Incorporated**

**Raleigh, NC**

# 46th Oilseed Conference

**Processing Efficiency:  
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**New Orleans, Louisiana, USA**

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## **WHOLE COTTONSEED RESEARCH & PROMOTION PROGRAM**

### **AT COTTON INCORPORATED**

**T.C. Wedegaertner and W.F. Lalor**

**Cotton Incorporated**

**Raleigh, NC**

### **Introduction**

Over the past 15 years, the price of cottonseed has averaged \$99 per ton at the grower level. During this same time period the total gross revenue received by growers for their cottonseed was \$567 million and cottonseed production averaged 5.7 million tons. Grower's revenue from seed was at a maximum during those years when production was close to 6 million tons. In 1991 the cottonseed crop hit a record of 7 million tons and the infrastructure in place to handle such a large crop was inadequate. Cottonseed storage facilities overflowed and the price received by growers plummeted to \$71 per ton and gross revenue fell to less than \$500 million (Table 1). This price drop was most severe in the Eastern U.S. where few oil mills existed and seed storage was insufficient. Some growers in this part of the Cotton belt received less than \$45 per ton for their seed in 1991. Since the demand for cottonseed appeared to be grossly inadequate to consume a 7 million ton crop at decent prices, cotton growers decided to spend a small portion of their check-off funds to stimulate demand for cottonseed. This became especially important when most forecasters were predicting cotton crops in excess of 20 million bales (8 million tons cottonseed) for the foreseeable future. The goal of the cottonseed program at Cotton Incorporated is to increase the value of cottonseed through research and marketing.

### **Cottonseed Marketing**

In recent years, a little more than half the cottonseed crop has been processed into value-added products by the crushing industry and slightly less than half was fed directly to livestock (mostly dairy cattle) without further processing. Marketing research, conducted by Cotton Incorporated,

determined that cottonseed has been available at a price that is less than its true feed value in most areas of the county where large concentrations of dairy cattle exist. In addition, it was also determined that less than 30% of the dairy feed market had been penetrated by cottonseed, so tremendous potential existed for increasing demand in that market segment for cottonseed. A complete marketing and promotion campaign has been put in place that includes print and radio advertising, trade shows, publicity and direct mail. The primary target of this effort continues to be dairy producers. The feed industry, dairy nutritionists and beef producers are also targeted.

### **Cottonseed Research**

Cotton Incorporated's research strategy to increase the value of cottonseed is divided into projects that are long term (10 years) and those that are short term (less than 5 years). The cornerstone of the long-term research strategy is the effort to remove gossypol from the seed. The objective is to eliminate gossypol from the seed while leaving it elsewhere in the plant where it acts as an insect deterrent. This research effort is now well on its way to accomplishing this objective.

Roasted cottonseed has been evaluated as a value-added feed ingredient for dairy cattle. A complete review of that research has been published (Wedegaertner and Lalor). The conclusion of that research effort was that cottonseed could indeed be heat treated in such a way as to increase the proportion of undegradable intake protein (UIP). The ideal roasting conditions were determined to be 146°C (295°F) for 30 minutes. The milk production response from cows fed the heat-treated cottonseed has been inconsistent at best. One study showed a trend towards increased production during mid-lactation, but the difference was not significant ( $p > .05$ ). Two feeding trials also investigated the blood gossypol response when roasted cottonseed is

substituted for unroasted seed. A consistent response of increased plasma gossypol was observed when heat-treated seed was added to the diet. This increased plasma gossypol was not of great concern except where other gossypol-containing ingredients, such as cottonseed meal and hulls, were also in the diet. No further work on roasted cottonseed is planned until more is known about the effects of heat treatment on gossypol availability.

The difficult handling and storage characteristics of cottonseed greatly limit its use in the feed trade. Several research projects have been conducted on cottonseed storage. A project in Mississippi (Willcutt and To) is investigating the engineering aspects of traditional storage systems. Lateral forces that are exerted by a cottonseed pile have been investigated in the hope that this information will prevent any further seed house failures. Air flow requirements and moisture migration are also being studied.

Alternative storage systems that could serve as emergency overflow or as storage for poor quality seed are being evaluated in Georgia (Bader, et al.). Silage bags appear to be suitable for storing good quality seed for extended periods, but the use of a common grain preservative did not seem to have any measurable effect on seed quality.

A simple, inexpensive system for putting a light starch coating on cottonseed to improve its handling characteristics has been developed (Laird, et al.). The process involves applying a hot, gelatinized cornstarch solution to fuzzy cottonseed, mix and then drying it in a belt conveyor dryer. The product, known as EasiFlo Cottonseed™, has been tested by several feed mills and it apparently meets their requirements for flowability. In addition to improved handling

characteristics, the coated product has approximately a 25% increase in bulk density, compared to fuzzy cottonseed. Research is underway to evaluate its shelf life and storage characteristics.

### **Conclusion**

Since the goal of Cotton Incorporated's cottonseed program is to increase the value of cottonseed, an evaluation of the program's success must consider the price of cottonseed and the total gross revenue received by growers for their seed. Figure 1 shows the five-year running average for the per ton price of cottonseed at the grower level. The five-year average is used to eliminate year-to-year fluctuations caused by various supply/demand forces. Note that during the period 1986 to 1989, cottonseed prices averaged about \$90 per ton. From 1990 to 1995, the per ton price seemed to plateau at about \$100. The effects of Cotton Incorporated's marketing program should be measurable within five years (1997). It is hoped that the increase in the average price in 1996 to near \$110 per ton is the beginning of another period of a relatively higher price for cottonseed.

Figure 2 shows the three-year average gross revenue received by cotton growers for their seed. A three-year average is used here since the natural forces of supply/demand tend to eliminate year to year fluctuations i.e., a large crop and low price or a small crop and high price. Note that the U.S. cottonseed crop was worth about \$400 million for the period 1984 to 1987. A gradual increase was observed over the next few years and it was not until 1993 that cottonseed revenue seemed to begin a steady increase. The cottonseed program at Cotton Incorporated will help contribute to a continued increase in cottonseed value.



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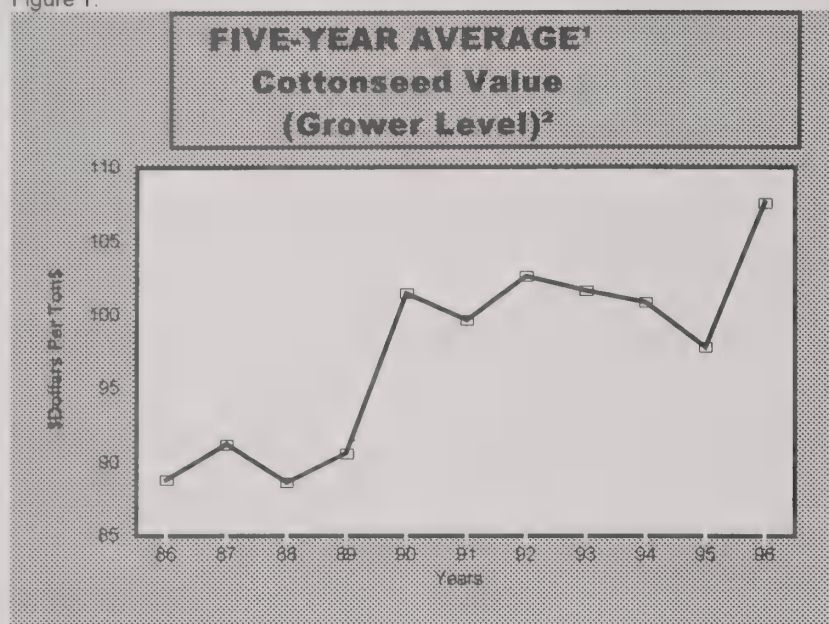
Table 1. Cottonseed Supply and Price

Crop year	Production Million Tons	Price Received by Growers \$/ton
82	4.7	71
83	3.1	131
84	5.1	95
85	5.3	67
86	3.8	80
87	5.8	83
88	6.1	118
89	4.7	105
90	6.0	121
91	6.9	71
92	6.2	98
93	6.3	113
94	7.6	101
95	6.8	106
96*	7.2	125

Source: USDA Cottonseed Update

\*Estimate

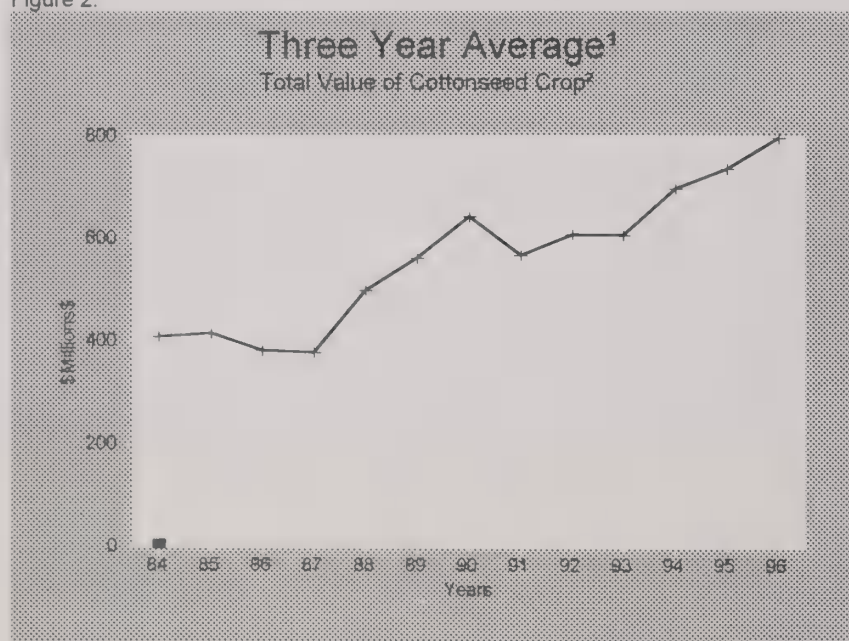
Figure 1.



<sup>1</sup>Five-year running average

<sup>2</sup>Source: USDA Cottonseed Update based on price paid the grower

Figure 2.



<sup>1</sup>Three-year running average

<sup>2</sup>Source: USDA Cottonseed Update based on price paid the grower

*New Technology Development - Genetics*

**Phil Kerr**  
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## **New Technology Development - Genetics**

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### **ABSTRACT**

Genetics has the potential to enable the development of products from oilseeds with modified composition. Conventional breeding techniques as well as molecular biology have been used in a complementary fashion to create soybeans with novel protein, oil and carbohydrate composition. The commercialization of novel processed protein and oil products will be facilitated by the development of grain and processed product logistic systems that preserve the identity of the products during manufacture and delivery. Application of these technologies for the development of improved animal feed, human food and vegetable oil products will be described. Examples will be presented of how this is being done with soybeans that possess increased lysine, sucrose or oleic acid content.

Just as chemical and engineering technologies have been successfully used to add value to oilseeds, genetics now has the potential to enable the commercial development of added value products from oilseeds, including soybean. Conventional breeding techniques like seed mutagenesis and germplasm screening as well as molecular biology techniques are being used to create soybean varieties with novel seed compositions. These techniques can also be used in a complementary fashion to increase the rate of commercialization of these varieties. These parallel research approaches use commercial targets derived from market research to develop commercial grain and processed grain products. The successful commercialization of novel processed products from these varieties will be facilitated by the development of grain and grain product logistic systems that can preserve the identity of the products throughout the channels of commerce. This presentation will provide examples of products from soybeans that are being developed using genetics which result in grain with modified protein, carbohydrate or oil composition.

The use of soybean meal in swine and poultry feeds has been reduced through the use of synthetic amino acids. In particular, inclusion of lysine and/or methionine in feed formulas can result in reductions of soybean meal of almost 30% compared to diets that do not utilize synthetic amino acids. Further, the use of alternative sources of protein from either animal (e.g., meat and bone meal) and plant (corn gluten) sources can limit the use of soybean in animal feeds. In addition, the substantial growth of the canola industry has limited the growth in utilization of soybean oil in various food applications. As a result, the United Soybean Board has identified these two sources of competition as being indicators of areas for improving the composition of soybeans. As a result, opportunities to improve soy protein products (e.g., meals, flours, concentrates and isolates) may be achieved by increasing the nutrient (e.g., amino acids, energy) density of soybean meal, as well as improving the functionality and nutrition of protein products used in human foods. In addition, opportunities have been identified to improve soybean oil by increasing its oxidative stability and modifying its saturated fat content.

At DuPont, three parallel approaches are being used to enhance the protein quality of soybeans: modification of amino acid biosynthetic pathways, expression of methionine rich zeins from maize and expression of lysine and methionine rich storage proteins. The pathway of aspartate derived amino acids (e.g., lysine, methionine and threonine) in developing seeds has been manipulated to increase the accumulation of lysine by expressing key regulatory enzymes which are not sensitive to allosteric inhibition by lysine. As a result, the lysine content of soybean can be increased by more than 2 fold. Production of desolventized, toasted soybean meals and subsequent *in vitro* analysis of available lysine has indicated that the increased lysine in high lysine soybeans processed under optimum pilot plant conditions is digestible. Results obtained from using the precision-fed rooster bioassay are consistent with those from the *in vitro* analysis. By expressing a methionine rich zein from maize, soybeans with a seed methionine content up to 75% higher than typical soybeans have been developed. Future research needs to be conducted to determine how high methionine soybeans behave during conventional processing as well as the bioavailability of the increased sulfur amino acids.

Targets for developing improved soy protein products (flours, concentrates and isolates) include reduced raffinose saccharide (e.g., stachyose and raffinose) content, increased sucrose content, improved flavor and increased isoflavone content. DuPont has successfully used conventional breeding techniques to modify the soluble carbohydrate biosynthetic pathway and develop soybeans with high sucrose and low raffinose saccharide content. Soy flour made from high sucrose and low stachyose grain can be used in a new process to manufacture a novel product. The benefits of the new process and the compositional features and benefits of the new protein product compared to functional soy concentrate and soy protein isolate include improved flavor, increased protein solubility and significantly higher total isoflavone content. Benefits of the new process include reduced waste product generation and improved efficiency of protein recovery.

DuPont has successfully used two strategies to modify the fatty acid composition of soybean oil. The first is the conventional plant breeding technique of seed mutagenesis and the second is the use of molecular biology techniques to control the expression of key fatty acid biosynthetic genes. In particular, the expression of oleoyl (D12) desaturase has been controlled in developing seed to create soybeans with oil having an oleic acid content greater than 80% of total fatty acids. The high oleic acid trait is also associated with reduced linoleic, linolenic acid and saturated fat content. In addition, the expression of acyl-ACP thioesterase has been controlled to produce soybeans with oil having a substantially reduced saturated fat content.

Fatty acid profile from high oleic soybean varieties grown in different locations in 1995 and 1996 has been highly stable among environments with oleic acid contents of approximately 82%. High oleic acid soybeans have been used to develop nondestructive methods to determine oil quality. These methods use near infrared reflectance spectroscopy. Oleic acid composition of soybean grain can be estimated nondestructively within 2 minutes with a precision of about 2% using this method. The results indicate that it is possible to accurately monitor the fatty acid composition of high oleic soybean through commercial grain production channels. The method should also prove useful to preserve the identity of the grain and processed oils during the extraction and refining processes.

Refined, bleached and deodorized oil from high oleic soybeans displays increased oxidative stability compared to a number of chemically processed oils. High oleic soybean oil also displays superior oxidative stability compared to oils from other genetically modified oilseeds including soybean, canola and sunflower. Collectively, the results indicate that oil from high oleic soybeans displays significant functional and compositional advantages compared to other vegetable oils. As a result, the efficiency of manufacturing high stability vegetable oils may be improved significantly. Efforts are underway to further evaluate high oleic soybean oil in a wide range of food and industrial product applications.



*Biotechnology in the Oilseed Industry*

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## **BIOTECHNOLOGY IN THE OILSEED INDUSTRY**

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### **ABSTRACT**

Engineering oilseeds for modified fatty acid composition or other traits transgenically or by plant breeding is a reality today, offering tremendous potential as well as complications for the oil and food industries. The wide array of designer vegetable oils has evolved over the past 40 years; however, the most rapid progress has occurred in the last 10 years. Conventional breeding methods have produced high-stability oils such as low linolenic acid soybean and high oleic/low linolenic acid canola. Low linolenic acid canola and soybean oils; high oleic sunflower, safflower and canola oils are commercially available in the U.S. Additional oils include high oleic corn and soybean, high palmitic and high stearic soybean, and low linolenic flax. Modified oilseed varieties have significant advantages such as providing an oil with a combination of low saturated fat and high stability without hydrogenation. Oils modified to have low linolenic acid or high oleic acid have longer fry life and foods fried in these oils have longer shelf life than the corresponding unmodified or non-hydrogenated oil.

Information about the effects of these modifications on food flavor quality and oil stability is needed to enhance utilization of new varieties as value-added alternatives to current oilseed cultivars. Oil with maximum stability during frying is a major objective of breeding and genetic engineering of oilseeds. As a result, plant breeders developed cultivars with high (85-90%) levels of oleic acid. Although this composition may contribute greater fry life, high levels of oleic acid may limit desirable fried food flavor intensity of fried foods. Research at our USDA Center has shown that fried food flavor intensity decreases with increasing oleic acid content. Cottonseed oil with its high (52-55%) linoleic acid content is considered the industry standard in the U.S. for producing fried food with desirable fried food flavor. On the other hand, foods such as potato chips fried in high linoleate-containing oils are not oxidatively stable. Optimum fried food flavor, oil fry life and shelf life of food need to be balanced. These modified oils are potential alternatives to hydrogenated oils especially in frying applications; however, optimum fatty acid compositions for each type of food application need to be determined.



## INTRODUCTION

Oils and fats intended for high stability applications such as frying must either be naturally stable or be stabilized to prevent deterioration. We can simply choose fats such as palm oil or tallow with fatty acid compositions lower in polyunsaturated fatty acids, higher in saturated fatty acids and higher in monounsaturated fatty acids. Modifying the fatty acid composition of the oil--the most common method to stabilize frying oils--can be done by several methods. For example, blending polyunsaturated oils with more saturated or monounsaturated ones is used to adjust fatty acid levels to optimum levels such as combining high oleic sunflower oil with corn oil or hydrogenated soybean oil with soybean oil (1-3). Chemically altering the existing fatty acid ratios by hydrogenation increases saturated fatty acids and decreases polyunsaturated fatty acids to produce more stable oil (3-4). A recent approach has been to modify fatty acid compositions of oilseeds by breeding to produce oils with greater frying stability, usually by decreasing linoleic acid and linolenic acid and increasing oleic acid (5). This approach began approximately 40 years ago, when genetic variants of safflower were selected in which the ratio of linoleic acid and oleic acid was reversed to produce high oleic safflower oil (6-8). During the past ten years, a variety of oils were developed with fatty acid compositions modified by plant breeding including low linolenic soy, high oleic sunflower, low linolenic canola, and high oleic canola (9-15). All these modified oils have improved frying stability compared to the unmodified oils.

This presentation is divided into the following segments: examples of modified oil; types and properties of conventional oils; properties of modified oil; applications of modified oil; advantages/limitations of modified oil; and potential of modified oil.

## TYPES OF OILS WITH MODIFIED COMPOSITION

Oilseeds with modified fatty acid compositions were first researched in the late 1940's; however, most of the developments have occurred in the last 10 years. A list of modified oils that are either commercially available or in development are in Table 1. Conventional breeding methods have developed high stability oils such as low linolenic acid soybean oil and high oleic/low linolenic canola oil. Low linolenic acid canola and soybean oils; high oleic sunflower, safflower and canola oils are now commercially available in the U.S. Comparisons of the fatty acid compositions of some modified and unmodified oils are presented in Figure 1. The levels of saturated fatty acids are not altered much between the two versions of each oil type; however, the most pronounced changes are decreased polyunsaturates and increased monounsaturates. Oils high levels of saturated fat are in commercial development. For example, high saturate soybean oil may provide functional benefits in food applications such as margarine and shortening base stocks without the need for hydrogenation. Plant breeders, food scientists and oil chemists are collaborating extensively to determine the end-use performance of modified oils and to select optimum fatty acid compositions for future oilseed cultivars.

**Table 1. Oils with Modified Fatty Acid Composition**

High Oleic Sunflower	High Oleic Corn
High Oleic Safflower	Low Linolenic Canola
Low Linolenic Soybean	High Oleic Canola
High Palmitic Soybean	High Lauric Canola
High Stearic Soybean	Low Linolenic Flax
High Oleic Soybean	Low Saturates Soybean



Modified composition oils are gaining attention because of the alternative they offer to the currently available polyunsaturated oils, tropical oils, animal fats and hydrogenated oil. Scientists discovered in the 1950's that the instability of some polyunsaturated oils was caused primarily by high linolenic acid levels (16,17). Proposed methods to remove linolenic acid included breeding it out; extracting it out; or reacting it out. During the 1950's, when these alternatives were first proposed, breeding out linolenic acid was not considered possible. Reacting out linolenic acid by hydrogenation was chosen as the most practical approach to produce a stable soybean oil with low (3%) linolenic acid for extended use at high cooking and frying temperatures in consumer and commercial markets. Modifying the fatty acid composition of linolenate-containing oil to improve frying stability has also focused on blending oils to decrease the linolenic acid or to decrease total polyunsaturated content (1-3). Frankel and co-workers (1) blended soybean oil with high oleic sunflower (Fig. 2) and found that peroxide values of aged oil blends decreased significantly as the ratio of high oleic sunflower to soybean oil increased.

## TRADITIONAL FATS AND OILS

Prior to the widespread use of polyunsaturated oils in the 1950's, foods were generally prepared with lard or tallow which were oxidatively stable and had good baking and/or frying characteristics. Cottonseed oil was also used extensively in the U.S. as a liquid frying oil. The trend toward polyunsaturated oils--corn, soybean, and sunflower--met a desire for lower saturated fat and no cholesterol; however, these oils did not have the stability of the more saturated fats. Soybean and canola oils have inherent disadvantages primarily because of high linolenic acid content which limits their use in frying and high temperature cooking. The inherent instabilities of fats and oils (Table 2) calculated from the relative rates of reactivity with oxygen for monounsaturated and polyunsaturated fatty acids shows significantly higher instability values for polyunsaturated oils than for saturated fats. High instability values correlate with decreased fry life of oil and shelf life of fried foods.

**Table 2. Inherent Instability of Fats and Oils Calculated from Relative Rates of Oxygen Reactivity**

<u>Oil</u>	<u>Iodine Value</u>	<u>Inherent Instability<sup>a</sup></u>
Safflower	149	7.6
Soybean	132	7.0
Sunflower	136	6.8
Corn	128	6.2
Low linolenic soybean	115	6.0
Canola	120	5.5
Cottonseed	110	5.4
Peanut	100	3.7
High oleic sunflower	85	1.9
Lard	62	1.7
Palm	50	1.3
Palm kernel	13	0.3
Coconut	8	0.2

<sup>a</sup>Based on relative rate of oxygen reactivity: oleic, 1; linoleic 10; and linolenic, 25

Modified from Erickson (18)

## PROPERTIES OF OILS WITH MODIFIED COMPOSITION

Genetic modification and breeding have changed fatty acids by decreasing linolenic acid and saturated fatty acids as well as by increasing oleic acid, palmitic, lauric or stearic acids. The impact of these alterations on vegetable oil flavor quality and stability and the overall oil composition is important in the effective utilization of new varieties as value-added alternatives to current oilseed cultivars. Now the results of extensive breeding programs are being realized and the end-use performance of oils from new cultivars with modified fatty acid composition are being studied. Soybean oils with less than 3% linolenic acid have improved high temperature stability relative to hydrogenated cooking oils and unmodified soybean oils (11-12). Researchers have also shown that decreasing linolenic acid levels increased the stability of soybean salad oils (19). Traditional genetic and plant breeding approaches have been used with success to modify oil composition in other oilseeds including sunflower, corn and rapeseed/canola with the primary emphasis on increasing oleic acid to high (85-90%) levels. Initially, the direction toward lower linolenic acid and higher oleic acid seemed appropriate since oils with these fatty acid profiles have greater frying stability as judged by less oxidation, polymerization and hydrolysis. Tests have shown that high oleic oils have good stability during high temperature heating and frying (6-8). However, at the U.S. Department of Agriculture's National Center for Agricultural Utilization Research (NCAUR) in Peoria, Illinois we have studied the effects of fatty acid composition on the fried food flavor intensity of fried foods and found that oils with high oleic acid levels do not produce fried food with desirable levels of fried food flavor intensity.

In a study using cottonseed oil, high oleic sunflower oil and blends of these two oils, we produced oils ranging in oleic acid content from 16% to 78% and with linoleic acid levels of 12% to 55%. Flavor analysis of fresh, unaged potato chips showed decreases in fried food flavor intensity and in overall flavor quality as the percent of oleic acid increased in the frying oils and as linoleic acid content decreased (Fig. 3). Potato chips fried in oil with 16% oleic/55% linoleic acid (cottonseed oil) had significantly higher intensity of fried potato flavor than potato chips fried in the oil with either 63% oleic/23% linoleic acid or 78% oleic/12% linoleic acid (high oleic sunflower oil). The effect of storage of the potato chips at 25°C for six months resulted in a different pattern of changes in fried potato flavor intensity and in overall flavor quality than in the unaged samples (Fig. 4). Potato chips fried in the oil with 42% oleic acid/37% linoleic acid had the highest fried food flavor intensities and flavor quality scores. Rancid flavor intensity was higher in the potato chips with the highest level of linoleic acid. These results agreed with those of Fuller *et al.* (7) who showed that potato chips fried in cottonseed oil had less flavor stability than potato chips fried in high oleic safflower oil. The fatty acid composition of the oils significantly affected the intensity of the fried food flavor in the potato chips as they were aged up to six months at 25°C (Fig. 5). Since few significant differences were noted in the fried food flavor intensity of potato chips sampled at 3, 6, 12, and 15 hr, the data were pooled over frying time. Plotting the means of the pooled data for fried flavor intensity showed that as the amount of aging increased from 0 time to 6 months, the fried food flavor intensity decreased. Initially, the samples fried in cottonseed oil with 16% oleic/55% linoleic acid content had significantly greater intensities of fried food flavor than potato chips fried in oils with 42% oleic/37% linoleic acid. The relationship of decreasing fried food flavor intensity with increasing oleic acid content was apparent at 0, 1 and 2 months of storage at 25°C. However, as aging increased, the higher intensities of rancid flavor possibly masked the fried flavor of the potato chips fried in oils with higher levels of linoleic acid. The sample fried in



oil with 42% oleic/37%linoleic acid had the highest level of fried food flavor by the 6 month storage time.

Potato chips fried in HOSUN had the lowest intensities of fried food flavor (Fig. 5). 2,4-decadienal, a breakdown product of linoleic acid oxidation has been reported to contribute to the characteristic fried flavor intensity (20). Therefore, 2,4-decadienal levels in the potato chips could possibly explain the correlation between the decreasing intensity of fried food flavor in the potato chips with the decreasing levels of linoleic acid in oils. The levels of 2,4-decadienal in fresh and aged potato chips showed that the isomers of this compound decreased significantly with increasing oleic acid content of the oil (Fig.6). The levels of 2,4-decadienal were significantly higher in the aged potato chips than in the fresh samples. 2,4-decadienal has also been reported as a good marker measuring for oxidative stability (21). The effect of this compound on the fried flavor intensity of the aged potato chips is complicated by the reports that it may be partly responsible for fried flavor as well as being a marker for lipid oxidation.

In other studies at NCAUR with low linolenic acid soybean oils, we compared soybean oil containing 7.7% linolenic acid and a commercial hydrogenated-winterized soybean oil (3% linolenic acid) with three samples of soybean oil with 3-5% linolenic acid (19). Results indicated there were few significant difference in flavor or oxidative stability during 60°C storage between modified and control oils. However, in high temperature stability tests at 190°C, low linolenic acid oils had improved overall room odor intensity scores and lacked the fishy odors of non-hydrogenated soybean oil and the hydrogenation odors of hydrogenated commercial cooking oil. In other tests with low linolenic acid soy oils, these modified oils also performed better as frying oils than as salad oils, probably because the unmodified oil is much less stable as a frying oil than as a salad oil.

In tests at NCAUR with high oleic corn oil (9% C16:0, 2% C18:0, 64% C18:1, 23% C18:2, and 0.6% C18:3) developed by Du Pont, we found that dry milled high oleic corn oil had significantly better storage stability as a salad oil than did unmodified corn oil (22) (Fig. 7). The reversal of the ratio of oleic to linoleic acid from corn oil to high oleic corn not only improved the oxidative and flavor stability characteristics of the modified oil but also its frying characteristics. High oleic corn oil had better frying stability than corn oil as measured by total polar compound formation (Fig. 8). Sensory analysis of the room odor characteristics of the heated oil showed that the dry milled and wet milled high oleic corn oils had significantly less overall odor intensity than unmodified corn oil or hydrogenated corn oil (Fig 9). The fried food flavor intensity of french fried potatoes was higher in samples fried in corn oil than in the high oleic corn oils or hydrogenated corn oil in the early stages of frying (Fig. 10). However, after 17.5 hr of frying, the high oleic oils had increased levels of fried food flavor intensity compared to the 9.5 hr sampling time. Generally, foods fried in high oleic oils show increasing fried food flavor intensity with increasing oil usage (11, 13, 23).

## **APPLICATIONS FOR OILS WITH MODIFIED COMPOSITION**

Oils with modified fatty acid composition can be used for a variety of foods in which stability of the oil or the shelf life of the fried food is a concern (Table 3). Deep fat frying which is commonly used in restaurants and in the snack food industry accounts for much of the market for cooking and frying oils. However, the chemical and physical processes (hydrolysis, polymerization and thermal oxidation) that occur during frying require stable fats and oils that are resistant to these

processes. Oils with modified fatty acid composition provide an excellent alternative to animal fats, tropical fats and hydrogenated oils. Spray oil applications can benefit from the use of modified oils that are stable under shelf life conditions. Other food uses of modified oils include base stocks for margarine and shortening and for non-dairy creamers. Numerous non-food applications include cosmetics, inks and lubricants.

**Table 3. Food and Non-food Uses of Modified Composition Oils**

<u>Food Uses</u>	<u>Non-food Uses</u>
Frying Oil	Cosmetics
Spray Oil	Printing Ink
Margarine	Lubricants
Shortening	
Non-dairy Creamer	

### **ADVANTAGES AND LIMITATIONS OF OILS WITH MODIFIED COMPOSITION**

Modified oilseed varieties provide us with oils that have significant advantages and potential limitations (Table 4). From a health aspect, some of the oils provide a combination of low saturated fat and high stability without hydrogenation. Oils modified to have low linolenic acid or high oleic acid have longer fry life and foods fried in these oils have a longer shelf life than the corresponding unmodified oil. In addition, foods produced from these modified oils do not contain positional and geometric isomers of fatty acids produced by catalytic hydrogenation. The polyunsaturated fatty acids, especially linolenic acid that contribute to strong off-odors such as acrid, fishy and burnt during frying are decreased to low enough levels in some of the modified oils so that these characteristic odors are not a problem. Unfortunately, modified oils are not produced in large enough quantities to satisfy the demand for frying and cooking oils--a 6 million pound market per year in the U.S. Also better systems to allow for identity preservation of each oilseed type are necessary.

**Table 4. Advantages and Limitations of Modified Composition Oils**

<u>Advantages</u>	<u>Limitations</u>
Lower saturated fat	Low production volume
No hydrogenation	Identity preservation
Longer shelf life	Low intensity of fried food flavor
Longer fry life	Cost
Less frying odor	Decreased omega-3 fatty acid
Less off-flavors	



## POTENTIAL OF MODIFIED COMPOSITION OILS

Although property enhanced oils are potential alternatives to traditional oils and fats, more research is needed to determine optimum fatty acid compositions necessary for each type of food application. Proposed compositions for frying oils given in Table 5 are presented from three different viewpoints--a snack food producer, oil processor, and food scientist. All three agree that saturated fats and linolenic acid should be low; however, the levels of oleic and linoleic acid each range over approximately 30%. Of course, stability tends to increase with increasing oleic acid and decreasing linoleic acid; but the flavor of the fried food is significantly affected by the level of linoleic acid. Low levels of linoleic acid tend to produce food with less fried food flavor. On the other hand, foods such as potato chips that are fried in high linoleate-containing oils are not oxidatively stable. A balance between optimum fried food flavor and fry life of the oil with shelf life of food needs to be determined.

**Table 5. Proposed Optimum Fatty Acid Compositions for Frying Oil**

	<u>C16:0</u>	<u>C18:0</u>	<u>C18:1</u>	<u>C18:2</u>	<u>C18:3</u>
Snack Food Producer	4	2	56	30	3
Oil Producer	4	2	90	3	1
Food Scientist	4	2	70	20	3

What are the issues that modified oils will face in the future (Table 6)? Economic and public perception issues include negative impressions of transgenically modified food; techniques to ensure identity preservation of the seeds; and cost/benefit ratio of modified oils to unmodified ones. Food manufacturers will be concerned with determining if oils should be modified for every type of use (designer oils) or if a few modified oils will be blended with unmodified oils to create desired compositions. Will modified oils be so widely accepted that they in turn become the commodity oil that they originated from? Finally, what will plant breeders be developing in the future--specific fatty acid compositions, stacked traits, and/or optimum tocopherol levels and ratios? Current and future end-use performance research will provide important information for plant breeders to develop new cultivars for food oils. The potential exists for a designer oil with a specific fatty acid composition for every food use; however, the practicality of this concept is a major issue to be resolved.

**Table 6. Future Issues for Genetically Modified Oils**

-Transgenic Issue	-Modified tocopherol content
-Identity Preservation	-Increased linolenic acid
-Commodity Oil	-Stacking of traits: high oleic, low linolenic, low saturates
-Designer Oils vs. Blending	-Optimum fatty acid compositions
-Cost	

## Figure legends

Figure 1. Fatty acid composition expressed as % polyunsaturated fatty acids (PUFA), monounsaturated fatty acids (MONO) and saturated fatty acids (SAT) of common unmodified oils with corresponding oil with modified composition

Figure 2. Peroxide values for soybean (SBO), high oleic sunflower oil (HOSUN) and blends of SBO and HOSUN aged 0-14 days at 60°C (modified from Frankel et al. )(1).

Figure 3. Overall flavor quality scores and flavor intensity scores of fresh potato chips fried in oils used for 6 hours of frying at 190°C.

Figure 4. Overall flavor quality scores and flavor intensity scores of potato chips fried in oils used for 6 hours of frying at 190°C and aged 6 Months at 25°C.

Figure 5. Fried food flavor intensity scores of potato chips fried in oils used for 6 hours of frying at 190°C and aged 0 to 6 Months at 25°C.

Figure 6. Relationship of 2,4-decadienal levels (left axis) and fried food flavor intensity scores (right axis/line chart) of fresh potato chips fried in oils used for 6 hours of frying at 190°C.

Figure 7. Flavor intensity scores and peroxide values for dry milled corn oil and dry milled high oleic corn oil aged 0-8 days at 60°C.

Figure 8. Total polar compounds in fresh and deteriorated oils used to fry french fried potatoes.

Figure 9. Room odor intensity scores for unmodified and modified corn oils after 1 hr of heating at 190°C.

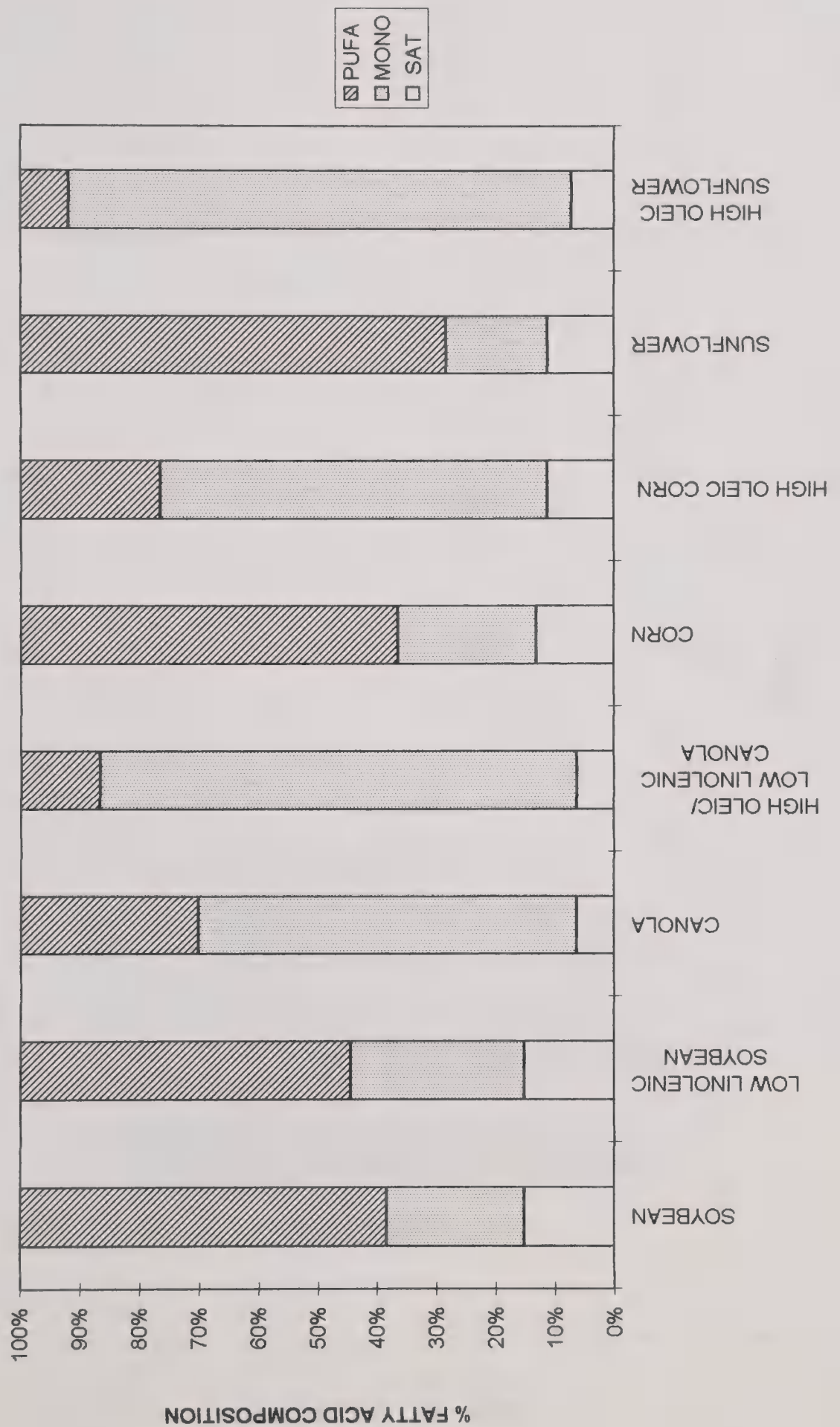
Figure 10. Flavor intensity scores for french fried potatoes fried in unmodified and modified corn oils after 9.5 and 17.5 hr of frying at 190°C.

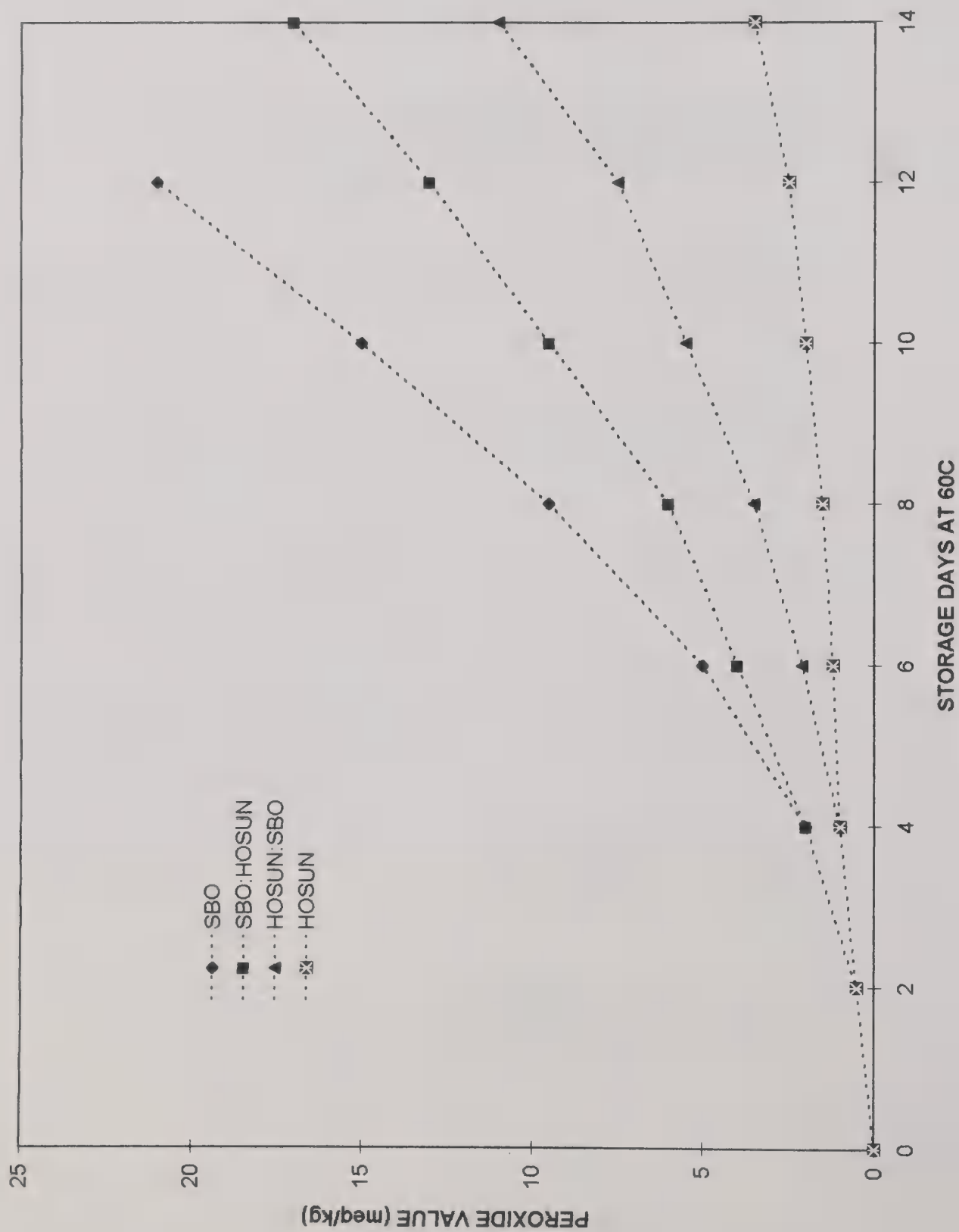
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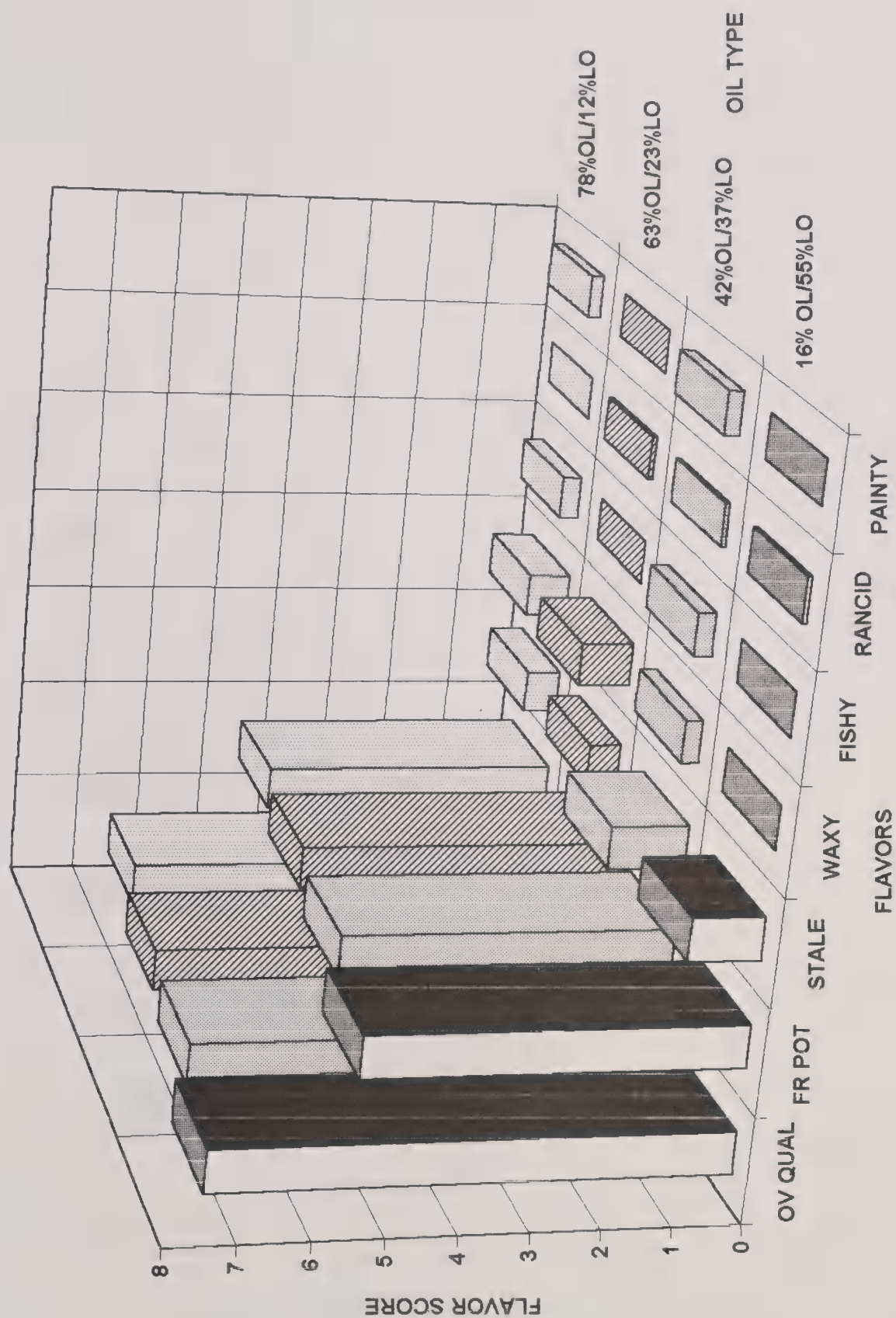
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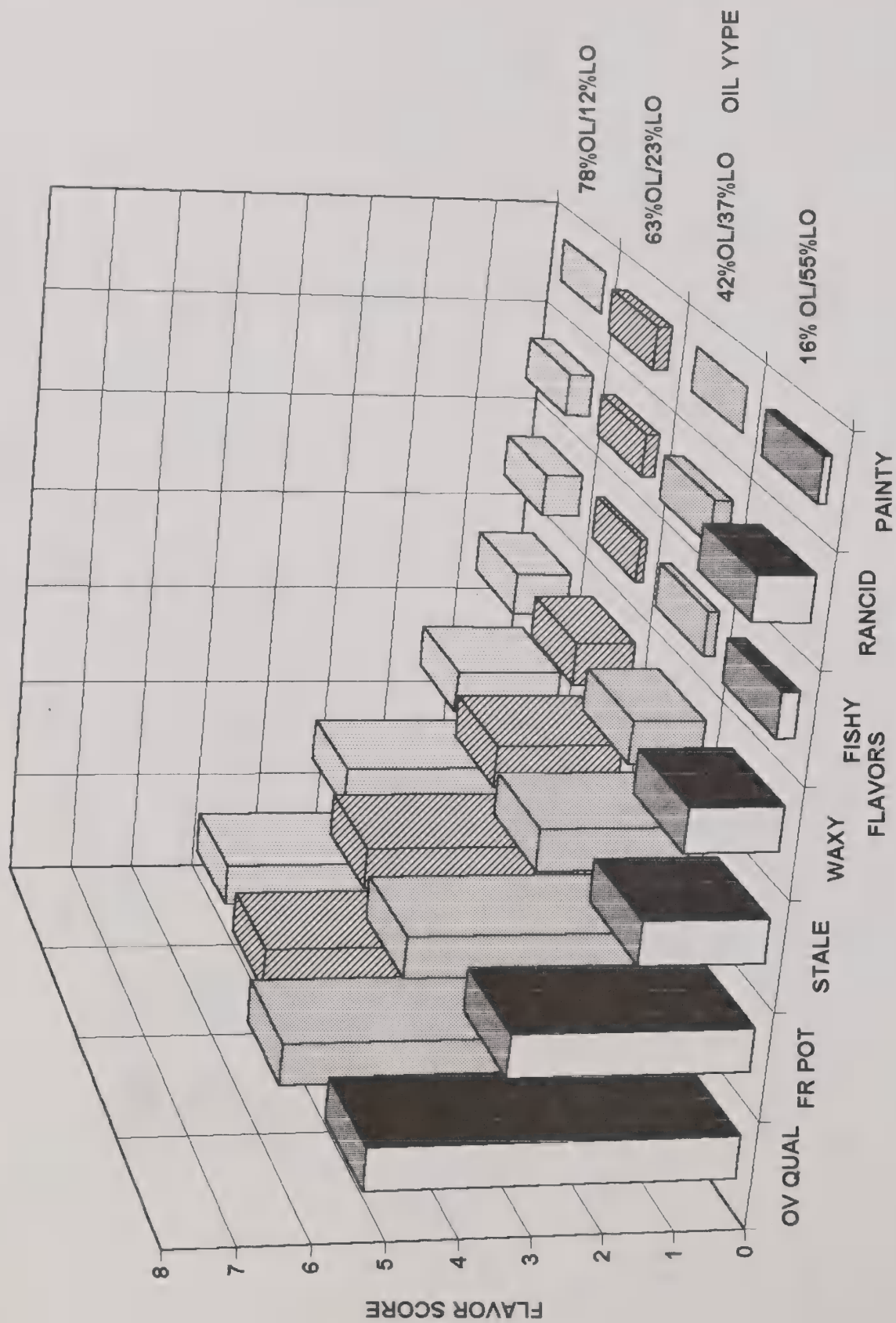
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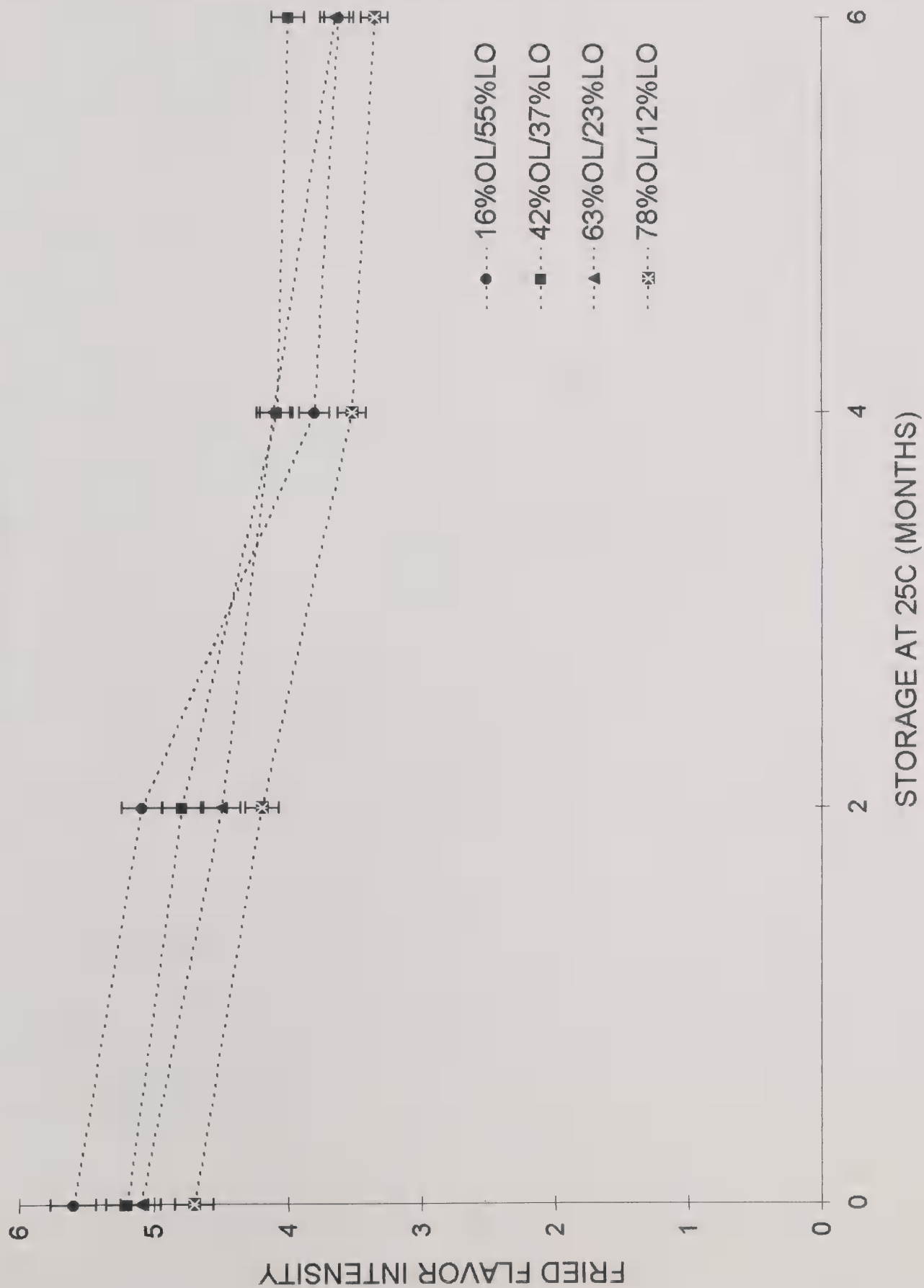


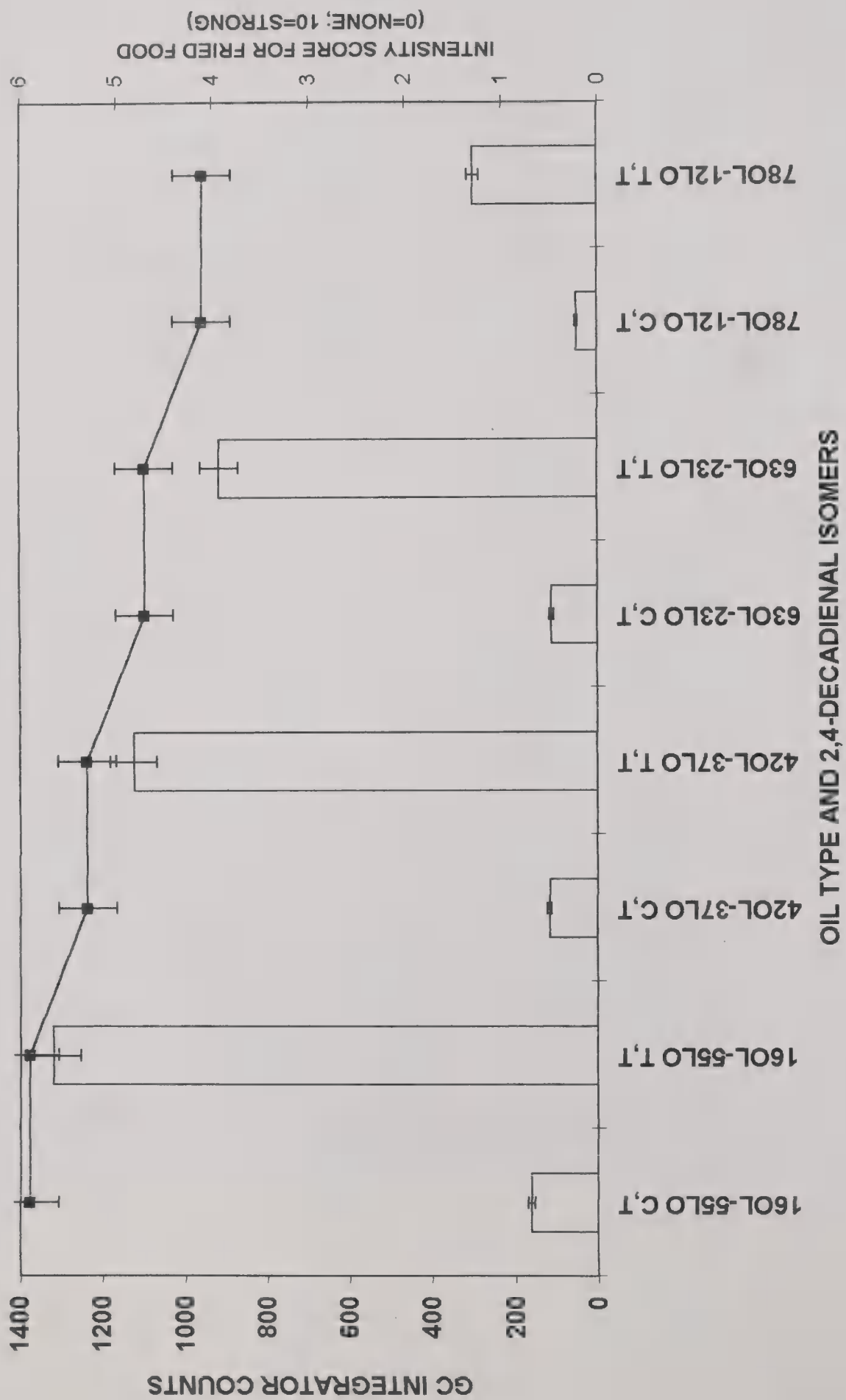


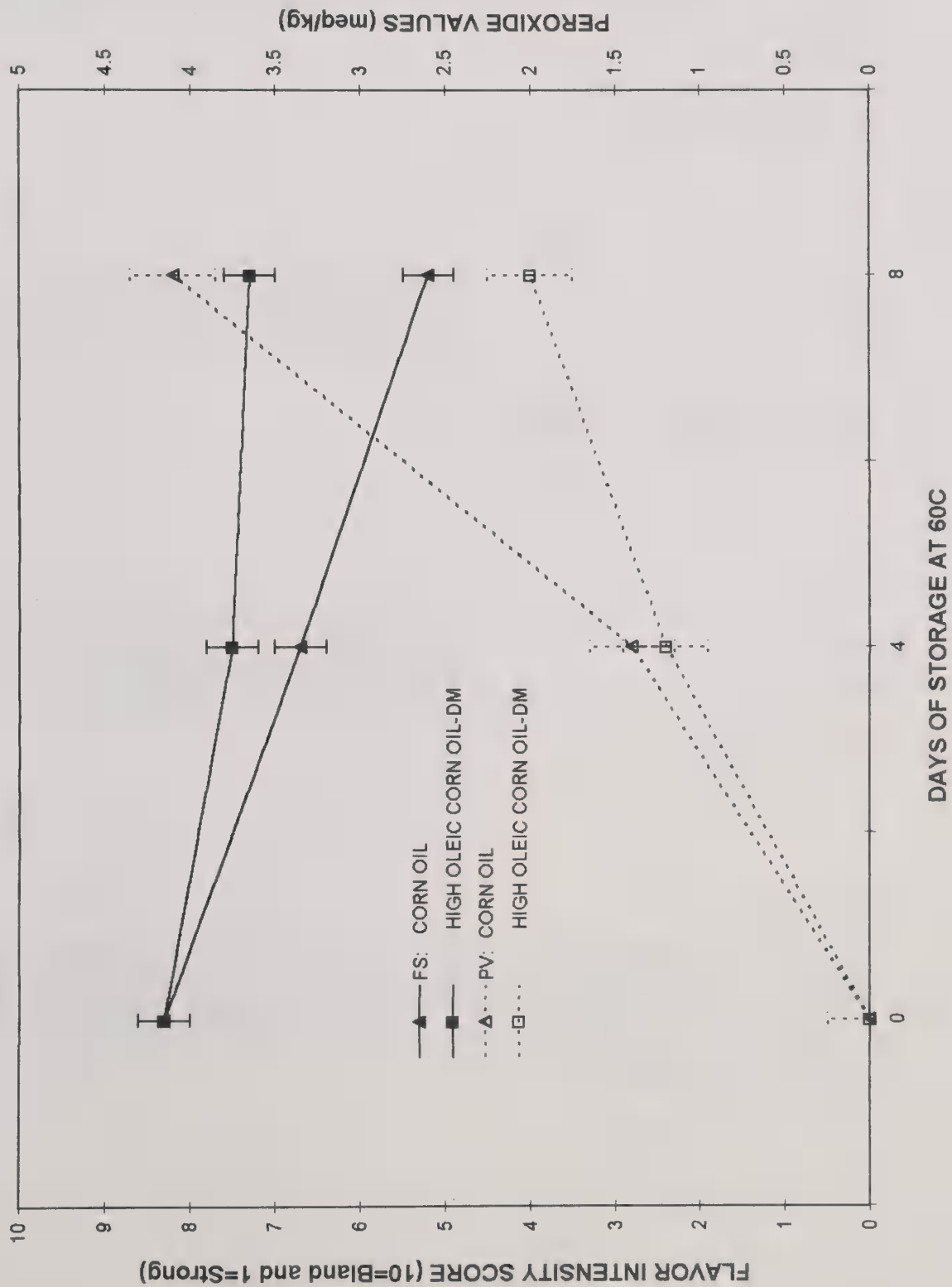


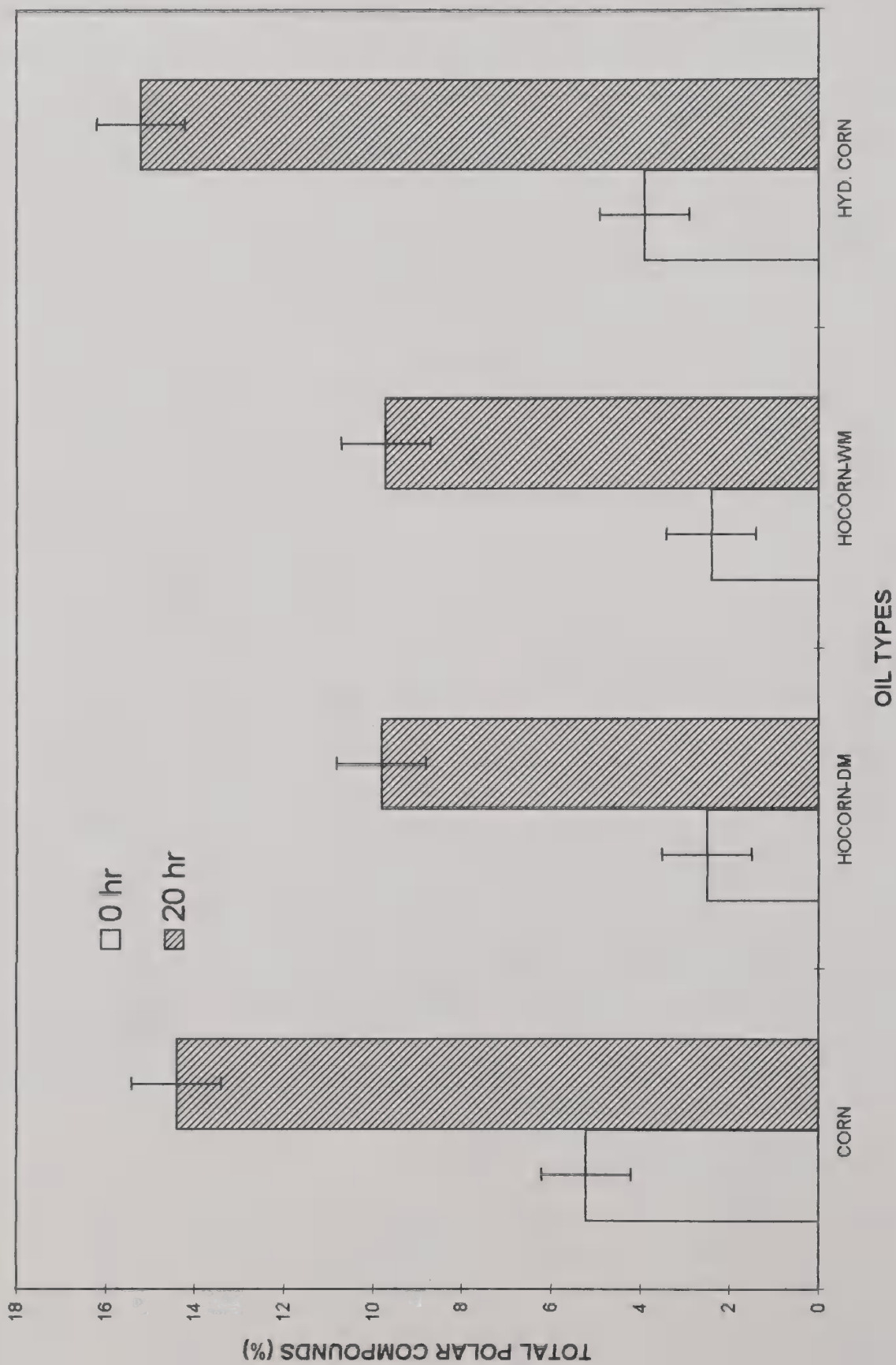














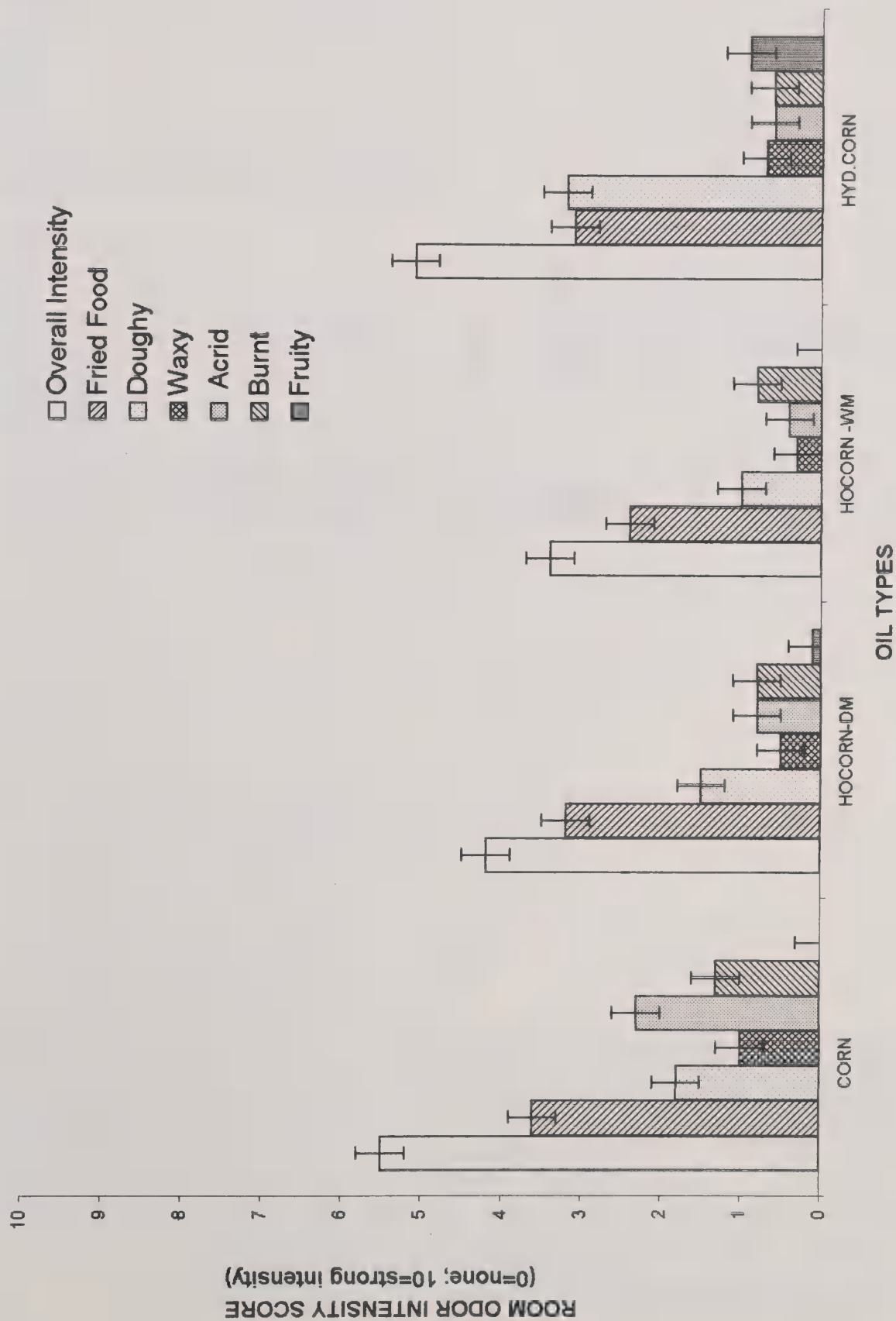
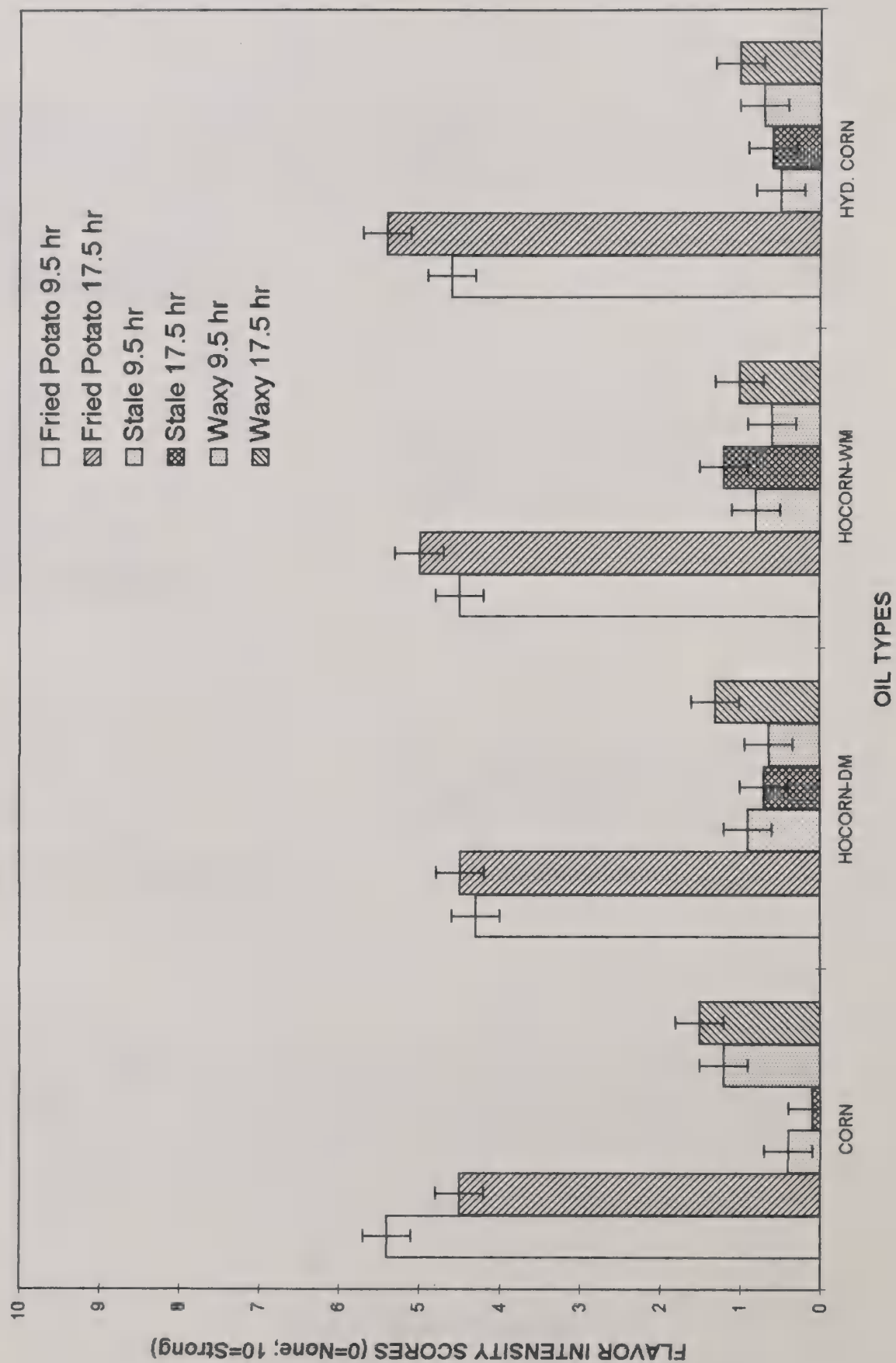


Fig 9



**Food Oil Substitutes - Frying Applications**

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**Urbana, IL**

# **46th Oilseed Conference**

**Processing Efficiency:  
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## Food Oil Substitutes - frying applications.

The fat and oil industry has undergone some interesting changes in the last few years. Two of the more important developments include the biotechnology based improvements to oilseed crops, and the development of non-caloric, fat-based, fat substitutes. A substantial amount of research is underway on the effect of lipids and lipid components on human health. More and more evidence accumulates that our health is at least partially dependent upon the fat and oils we consume, including the trace components in the oil.

Finally, a fat-based fat substitute, Olestra, has been approved by the U.S. Food and Drug Administration (FDA). It is marketed as Olean™ by Procter and Gamble (Cincinnati, OH). Some companies, particularly Procter and Gamble (P & G), were beginning to think that approval would never happen. Although P & G did alter the composition of olestra to minimize the laxative effect, the FDA has required the manufacturer to add a warning to the label about the potential gastro-intestinal problems. The FDA has also required the addition of the fat soluble vitamins A, D, E and K to any food product containing the fat substitute, since it can inhibit or reduce the absorption (10-20%) of the fat-soluble vitamins, including the carotenoids (1).  $\beta$ -carotene, as well as other carotenoids, can have substantial *in-vivo* anti-oxidant activity. The role of carotenoids in human health is still not well understood and research continues in this area.

The safety of the fat substitute was the primary concern of the FDA, just like all food additives. The potential gastrointestinal effects

do not have any medical consequences, although the labeling will provide the consumer with the needed information to determine whether they should discontinue the product, if necessary.

The FDA evaluated more than 150,000 pages of documents from more than 150 studies. In addition to these evaluations, the FDA sought advice from a special working group or subcommittee of experts from its Food Advisory Committee that met in November of 1995 to address these questions. The committee evaluated the material and data presented by the FDA, P & G, and other organizations and entities both opposing and supporting olestra's approval. After that evaluation, a clear majority of the working group agreed that all of the major safety issues had been identified and that the proposed use of olestra would be safe. A majority of the full Food Advisory Committee reaffirmed that judgment, after completion of their evaluation. The FDA will require the companies involved to monitor consumer reaction to the product.

Safety assessment is based on several important factors (2,3), which can be summarized as follows:

- 1) The exact chemical structure of the substitute must be known and documented; if it contains more than one compound the structure of each must be determined.

- 2) The stability should be well documented, especially during food production, storage and final preparation. If partial degradation occurs, the products should be identified. If any harmful impurities are present, concentration limits should be established.

3) Detailed exposure assessment for each population type should be included, which would include toxicological data demonstrating that the use of the macro nutrient substitute, as well as any of the degradative byproducts, would not cause harm to at least the levels of exposure for the intended use.

4) Additional tests may be required to provide pharmacokinetic, metabolic and/or nutritional data not normally required for other additives.

5) It will be necessary to determine whether the macro nutrient substitute is absorbed or not. If it is poorly absorbed, one needs to determine whether large concentrations effect the morphology, physiology, biochemistry or normal flora of the gastrointestinal tract.

6) The selection of animal models, experimental protocols and measurement techniques must be carefully planned to provide data, which can be used to accurately assess the safety of the substitute. In addition, consideration of the appropriate controls and control experiments would be advised.

7) Sufficient data must be provided to demonstrate that the substitute or its metabolites do not interfere with absorption or metabolism of essential nutrients. If interference does occur, one must determine whether it can be safely offset with nutrient fortification. If the compound is adsorbed at a measurable rate, there must be data indicating the impact, if any, on the nutrient status of the animal. In general, traditional assessments of nutritional status should be sufficient for nutrient status assessment.

8) Another area of special concern is the effect of macronutrient substitutes on selected, particularly sensitive, population segments, such as the very young, senior citizens or those afflicted with certain health problems.

9) The effect of the macronutrient on drug absorption and/or activity should be assessed. Some of the oil substitutes can have a slight laxative or anal leakage effect, at least during the initial stages of consumption until the digestive system has adapted to the product. Since this effect is often greater among children than adults, it may require additional evaluation. Special labeling may be needed to address the problem.

10) Post market surveillance will be needed in most cases. One long-term potential problem of particular concern could occur due to the consumption of several macronutrient substitutes concurrently, since it is likely that more than one of those materials may be approved in the future. There may be a synergistic or interactive effect on some of the factors just discussed. This concern is also likely to limit the number of fat-based fat substitutes that are eventually approved.

So now that one of the fat-based fat substitutes has overcome the huge approval hurdle, it is time to take the product group seriously, rather than just consider it as a academic exercise. How does this category of fat-substitutes fit in the overall product group of fat substitutes? There are four general categories of fat substitutes: the protein based fat mimetics, the carbohydrate (starch and fiber) based fat-mimetics, the reduced calorie triacylglycerols and the very-low to non-caloric fat-based fat substitutes. Olestra is



in the final category, the very-low to non-caloric fat-based fat substitutes.

Simplesse, derived from milk or egg protein, is the best known of the protein-based fat substitutes. The protein is particulated during a combined heating and homogenization process that produces small protein spheres of uniform size approximately 1  $\mu\text{m}$  in diameter (4). Simplesse has been affirmed as GRAS by the FDA for use in several products. The main limitation, which is the limitation for all currently approved fat substitutes and mimetics, except Olestra, is that it can not be used for many heated product applications, particularly frying.

There are several starch derived fat replacers or mimetics available (5), which are essentially maltodextrins. They are produced upon partial enzymatic or acid catalyzed hydrolysis of starch and are fully digestible. The low dextrose equivalent (DE) maltodextrins have fat binding functional properties, unlike the high DE products. Starches and sugars contain 4 kcals per gram. However, they are generally used as aqueous suspensions at concentrations much less than 100%, so the actual caloric content is usually 0.5 to 2 kcals/gram.

The fiber based products include gums, celluloses, hemicelluloses, pectins,  $\beta$ -glucans, and lignins, which are isolated from a variety of sources, including cereals, fruits, legumes, nuts and vegetables. Gums are typically not used as fat substitutes directly, rather they are used at low concentrations (0.1-0.5%) to form gels that increase product viscosity.

The third category is the reduced calorie fat-based fat substitutes. Caprenin is a reduced calorie triacylglycerol (Procter and Gamble) formed by the esterification of three fatty acids, caprylic, capric and behenic (6). The behenic acid is only partially absorbed, so caprenin contains 5, rather than the normal 9 kcal per gram. Caprenin has functional properties similar to cocoa butter and is intended to replace some of the cocoa butter in selected confectionery products. Another fabricated triacylglycerol, similar to caprenin, is Salatrim, which is comprised of a mixture of long chain (primarily stearic acid) and short chain (acetic, propionic and butyric) fatty acids randomly esterified to glycerol (7). It also contains approximately 5 kcal per gram (Nabisco Foods Group). It has the same utility that caprenin does as a fat replacer in reduced fat systems and could be used as a cocoa butter substitute in confectionery products and in baked products and filled dairy products. A third reduced calorie short chain triglyceride is Captrin from Stepan Food Ingredients (8). It is a randomized triglyceride made from linear saturated fatty acids primarily C8 to C10 in length. Proposed uses include baked goods, confections, dairy product analogs, snack foods and soft candy. All of these modified triacylglycerols are not useful for frying, because of excessive volatility at frying temperatures. In addition, release of the smaller molecular weight fatty acids may cause undesirable flavor notes in the food product.

The fourth group is the fat-based fat substitutes (9), which include fatty acids esterified to a glycerol substitute, such as a sugar or sugar alcohol, e.g., olestra or sorbestrin; or fatty acids attached to a

modified glycerol molecule, such as fatty acid esterified propoxylated glycerol; or fatty alcohols esterified to organic acids; or, finally, an alteration in the normal ester bond, such as reduction of a normal ester bond to an ether. Each of these modifications reduces the susceptibility of the molecule to the digestive enzyme lipase.

Without removal of the fatty acids, absorption will not take place and the caloric content is essentially zero. There is a synthetic fat substitute that contains neither fatty acids or fatty alcohols, termed polysiloxanes. It is not fat derived.

The development of fat substitutes is market driven, over 3/4 of U.S. consumers use low-fat and/or low-calorie foods. Companies are narrowly focused on developing products their customers will buy. Consumers continue to increase their concern about health issues and food, particularly with respect to fat content. The food industry has responded to consumer concerns and reduced the fat content, the cholesterol content and the amount of saturated fat in food products. Nutritionists continue to advise that consumers should reduce the percentage of fat in their diet, and increase the percentage of complex carbohydrates in their diet. Over the past decade several health organizations, including the American Heart Association and the Surgeon General, have recommended a reduction in total dietary fat to  $\leq 30\%$  of the total calories and  $\leq 10\%$  of total calories should come from saturated fat (10). A high intake of total dietary fat is associated with obesity, some types of cancer, and possible gall bladder disease. Diets high in saturated fat are associated with heart disease. However, consumers may not have

heard the message; fat and oil consumption increased from 60.4 lb/yr in 1985 to 65.0 lb/yr in 1993.

The three most important factors consumers consider when selecting food products are taste (84%), product safety (71%), and nutrition (69%) (11). The fourth factor was cost at a distant 42%. Consumers are indicating that they are reducing their consumption of red meats, dairy products and increasing their consumption of chicken, fish and high-fiber cereal. The majority of consumers (72%) when asked about olestra, were either interested or very interesting in purchasing a fried or baked product with olestra in which some of the fat was replaced.

Fat provides some important functions in food that make it very difficult to replace. Fats and oils affect food texture, flavor, mouthfeel, palatability, satiety, ingredient solubilization and transport and heat transfer properties during frying. Fats and oils contain essential fatty acids critical to good health (12, 13). In 1990 the market for fat substitutes surpassed \$100 million and has continued to grow. It could reach nearly \$7 billion for the U.S., Europe and Japan by the year 2000. The approval of olestra should enhance the growth potential.

There are other technologies under development that could reduce the consumption of fats and oils, while increasing consumption of fried foods. Nutrasweet has developed a gel based barrier that can be applied to the surface of a food product prior to frying that can reduce fat absorption by as much as 2/3. Their gellan gum provides a moisture barrier that retards fat absorption during frying. It works well with French fries and peanuts. It can



also be added to batters or breadings and used with chicken, fish, cheese, vegetables, and dough enrobed food items, such as egg and pizza rolls.

The approval and extensive use of fat-based fat substitutes is very unlikely to reduce fat and oil consumption in the U.S. A quick review of the chemistry of the fat-based fat substitutes reveals the reason. For example olestra, when fully esterified, contains nearly 8 fatty acid molecules per molecule of sucrose, while another potential fat substitute, glucose fatty acid methyl ester, contains four molecules of fatty acids per molecule of glucose. Normal triglycerides are derived (100%) from food fats and oils, while olestra contains fatty acids from food oils. Over 90% of Olestra, is derived from food oils. Fat-based fat substitutes are a win-win situation for oil seed processors, since the most likely source of fatty acid for fat-based fat substitutes will be oilseeds. It is certainly possible to make fat substitutes from animal fats and tropical fats. However, that competition should not be any different than the current situation.

There are advantages to fat-based fat substitutes that extend beyond a simple reduction in calories and fat content, although that alone is sufficient reason for many people to purchase the product. One can design a combination of olestra and regular fats or oils, in which the olestra contains primarily saturated fatty acids and the other fat or oil contains primarily non-hydrogenated, unsaturated fatty acids. This would allow a reduction in the percentage of saturated fat in the diet (as recommended by various health organizations). This would also allow production of an oil with an acceptable stability that would be directly related to the ratio of the two oils.

Fat replacers work, but not the same with all consumers. The use of fat substitutes can aid in the reduction of fat intake, but not necessarily overall energy intake. In a study of 24 lean, young men using olestra to replace fat in the breakfast meal, the total caloric content remained constant, while fat consumption was significantly reduced (14). However, a diet containing olestra can result in a reduction in both weight and the caloric content of the diet (1). Another advantage of olestra is that it can reduce cholesterol absorption, a property that was patented by Procter and Gamble. It is likely that the other poorly absorbed fat-derived fat substitutes will have a comparable effect.

There is also evidence that fat-based fat substitutes produce the same flavor compounds as does regular fats and oils (15, 16). Fried food flavor is one of the primary reason fried foods are so highly desired. Fat-based fat substitutes containing the appropriate unsaturated fatty acid (or fatty alcohol) should provide the desired flavor compounds, without the unwanted calories.

The approval of additional fat-based fat substitutes will probably be limited, at least initially, to certain commercial applications, which might exclude retail or home frying applications. During commercial production frying operations, oil absorption occurs at such a substantial rate that the removal rate due to absorption exceeds the degradation rate. The oil is never discarded, just continuously replenished during frying. In a retail establishment, the oil is used until substantial degradation has occurred, usually as indicated by excessive smoking. As a result, oil degradation products may accumulate to much greater

concentrations in the oil used in retail outlets, as compared to large-scale commercial frying operations. This may not be a problem, but it should be considered.

Children 2 years old or younger should rarely be placed on limited caloric content diets. Fat-substitutes are not appropriate for this age group, so they should probably be excluded from products that are consumed primarily by children and limited to products consumed primarily by adults. This would include savory snacks (currently approved), desert products for adults, entrees for adults, candy primarily for adults (e.g, gourmet chocolates), and selected processed meat products, which would sausages.

According to an analysis on the fat substitute industry by the Freedonia Group (Cleveland, OH) (not published), during the next decade consumers will continue to emphasis "healthy" food products, such as low or reduced fat food products. The biggest growth areas will be the fat-based fat substitutes, which includes olestra. The next largest growth area is the GRAS type fat-substitutes, which would include gums, starch, proteins, emulsifiers and combinations thereof. Another likely growth area is soy based products, which tend to be low fat, as well. Currently, there are nearly 300 fat substitute or replacer type products on the market. A growth rate of approximately 20% is expected through the year 2000. Market opportunities for the reduced fat and fat-substitute categories should increase and new fat-substitute products will result in increased product prices, although there will be some price pressure on established brands.

One area of considerable potential is the international market. Consumers in developed countries that recently undergone substantial economic changes, as well as consumers in developing countries that have enjoyed substantial success in recent years, may soon be in a position to afford expensive commodities and processed food products from the U.S. and Europe. Eventually, there will be a market for in fat-substitutes in those countries, although that is probably more than decade in the future. Opportunities for increased oilseed imports from the U.S. should be more immediate.



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*Rethinking Your Safety Program*

**Raymond Rush**

Safety and Industry Consultant

Jackson, MS

# 46th Oilseed Conference

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The National Cottonseed  
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## RETHINKING YOUR SAFETY PROGRAM

Author & Presentation by:

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THE ACCIDENTS AND INJURIES IN THE WORK PLACE DO NOT JUST HAPPEN. THEY ARE RESULTS OF UNSAFE ACTS OR CONDITIONS. MOST ACCIDENTS AND INJURIES ARE CAUSED BY UNSAFE ACTS. RATHER THAN UNSAFE CONDITIONS, IN ACCIDENTS ON THE JOB, ROUGHLY FOUR ARE CAUSED BY UNSAFE ACTS FOR EVERY ONE BY CONDITIONS.

THE ATTITUDE OF EMPLOYEES IS THEREFORE A KEY FACTOR IN ACHIEVING A BETTER SAFETY RECORD. GOOD MORALE RESULTS IN FEWER ACCIDENTS AND ILLNESS AND ENTHUSIASM TO RETURN TO WORK AFTER AN ACCIDENT.

AN EXAMPLE MIGHT BE THE OCCUPATIONAL SAFETY AND HEALTH ADMINISTRATION, CREATED IN 1970. SAFETY IN THE WORK PLACE WAS SURELY THE RIGHT MISSION FOR OSHA. I THINK WHEN OSHA REALIZED THAT SAFETY IN THE AMERICAN WORK PLACE HAD NOT IMPROVED GREATLY IN THE PAST TWENTY-FIVE YEARS, OSHA STARTED RETHINKING SAFETY IN THE WORK PLACE.

THERE MAY BE SLIGHTLY FEWER DISABLING INJURIES NOW THEN THERE WERE IN 1960 OR 1970, AND TO BE SURE, THE WORK FORCE HAS INCREASED

TREMENDOUSLY OVER THOSE YEARS. BUT WHEN YOU LOOK AT NEW AUTOMATION IN THE WORK PLACES THAT REPLACED EMPLOYEES IN SOME DANGEROUS MANUFACTURING JOBS. SAFETY IN THE WORK PLACE MAY ACTUALLY HAVE DETERIORATED SINCE 1970. WE NOW KNOW THAT WE HAVE BEEN GOING ABOUT THE TASK IN THE WRONG WAY. AT THAT TIME OSHA WAS RUNNING ON THE ASSUMPTION THAT AN UNSAFE ENVIRONMENT IS THE PRIMARY CAUSE OF ACCIDENTS, AND IT THEREFORE TRIED TO DO THE IMPOSSIBLE; CREATE A RISK-FREE UNIVERSE. OF COURSE ELIMINATING SAFETY HAZARDS WAS THE RIGHT THING TO DO. BUT IT IS ONLY ONE PART OF SAFETY AND PROBABLY THE LESSER PART. IN FACT, BY ITSELF IT ACHIEVES NEXT TO NOTHING.

THE ONLY EFFECTIVE WAY TO PRODUCE SAFETY IS TO ELIMINATE UNSAFE ACTS. OSHA'S DEFINITION OF AN ACCIDENT - "WHEN SOMEONE GETS HURT" IS INADEQUATE. TO CUT DOWN ON ACCIDENTS, THE DEFINITION HAS TO BE A "VIOLATION OF THE RULES OF SAFE BEHAVIOR", WHETHER ANYONE GETS HURT OR NOT. THAT'S WHEN WE START AND CONTINUE TO TRAIN EMPLOYEES TO WORK SAFE AND REMEMBER, "PRESENT MOMENT THINKING AND LOOK OUT FOR HIDDEN HAZARDS."

PETER F. DRUCKER'S ARTICLE "REALLY REINVENTING GOVERNMENT" SAID "THIS IS THE DEFINITION UNDER WHICH THE UNITED STATES HAS BEEN RUNNING ITS NUCLEAR SUBMARINES. ANYONE IN A NUCLEAR SUBMARINE, WHETHER THE COMMANDING OFFICER OR THE MOST JUNIOR SEAMAN, IS PUNISHED FOR THE SLIGHTEST VIOLATION OF THE RULES OF SAFE BEHAVIOR, EVEN IF NO ONE GETS HURT. AS A RESULT, THE NUCLEAR SUBMARINE HAS A SAFETY RECORD UNMATCHED BY ANY INDUSTRIAL PLANT OR MILITARY INSTALLATION IN THE WORLD: AND YET A MORE UNSAFE ENVIRONMENT THAN A CROWDED NUCLEAR SUB CAN HARDLY BE IMAGINED."

RETHINKING YOUR SAFETY PROGRAM, REMEMBER ENGINEERING SAFETY INTO WORK BY SAFETY DEVICES, EQUIPMENT, WHISTLES AND BELLS IS A COSTLY MATTER AND IN SOME CASES IS TRADED OFF. USING A COST BENEFIT ANALYSIS, WHEN THESE ENGINEERED SAFETY SOLUTIONS BECOME MORE COSTLY THAN "TRAINING" THE WORKERS, THIS CAN BE SHORT-SIGHTED THINKING. IT TAKES AN ENLIGHTENED MANAGEMENT TEAM TO UNDERSTAND THAT SAFETY IS A LONG-TERM INVESTMENT IN TERMS OF MANPOWER AND DOLLARS AND BOTH ENGINEERED SAFETY SOLUTIONS AND TRAINING ARE NECESSARY IN MANY SITUATIONS.

MOST OF US HAVE SEEN INSTANCES WHERE SUPERVISORS OR MANAGERS ARE UNWILLING TO OR DON'T WANT THE HASSLE OFTEN FORCING INFRACTIONS OF SAFETY RULES. THEY CAN'T BE BOTHERED TO CALL A

WORKER ON THE CARPET FOR PERFORMING AN UNSAFE ACT THAT DID NOT RESULT IN AN ACCIDENT OR THEY SIMPLY DO NOT HAVE A PROCEDURE FOR HANDLING INFRACTIONS OF SAFETY RULES BY WORKERS.

THE AVOIDANCE OF ENFORCING SAFETY RULES IS PROBABLY THE SINGLE MAJOR REASON WHY WORKERS CONTINUE TO PERFORM UNSAFE ACTS. UNSAFE ACTS THAT ARE NOT CORRECTED EITHER ALWAYS HAVE BEEN OR SOON BECOME NORMAL BEHAVIOR. IN MANY CASES UNSAFE ACTS ARE SILENTLY APPROVED OF BY MANAGEMENT SINCE NO ENFORCEMENT OF SAFE WORK PRACTICES TAKES PLACE. THE RESULT IS A CONFUSED WORKER BECAUSE MANAGEMENT PREACHES SAFETY AND IGNORES IT IN PRACTICE. NO LONGER CAN A SUPERVISOR BE LENIENT IN ALLOWING AN UNSAFE ACT TO CONTINUE. IF SAFETY GOGGLES ARE REQUIRED TO BE WORN, THEY MUST BE WORN.

NO LONGER CAN A SUPERVISOR TOLERATE CLUTTERED FLOORS, BLOCKED AISLES, EXTREME NOISE, HEAVY VIBRATIONS, INSUFFICIENT LIGHT, POOR VENTILATION OR DEFECTIVE MACHINERY AND TOOLS.

NO LONGER CAN A SUPERVISOR THINK SAFETY TRAINING AND OBEDIENCE TO SAFETY RULES WILL BE CONSIDERED "KID STUFF."



MANAGERS AND SUPERVISORS MUST WHILE COMMUNICATING TO EMPLOYEES ABOUT OPERATION OR SAFETY, BE A GOOD LISTENER. LEARNING THE FINE ART OF LISTENING TAKES PRACTICE. YOU MUST UNDERSTAND REAL LISTENING INVOLVES MORE THAN JUST HEARING THE SPOKEN WORD.

IN THE BOOK "YOUR ATTITUDE IS SHOWING" BY ELWOOD N. CHAPMAN, HE SAYS ATTITUDE IS A COMMON WORD. YOU HEAR IT ALMOST EVERYDAY. PROFESSORS USE IT ON CAMPUS, MANAGERS DISCUSS IT AT WORK, EMPLOYMENT COUNSELORS LOOK FOR IT AMONG APPLICANTS. NO OTHER ATTRIBUTE WILL HAVE MORE INFLUENCE UPON YOUR FUTURE. A POSITIVE ATTITUDE CAN BE YOUR MOST PRICELESS POSSESSION.

IF YOU CAN CREATE AND KEEP A POSITIVE ATTITUDE TOWARD YOUR JOB, YOUR COMPANY, YOUR SAFETY PROGRAM AND LIFE IN GENERAL, THEN AND ONLY THEN WILL YOU BE A GOOD SUPERVISOR OR LEADER.

A POSITIVE ATTITUDE IS ESSENTIAL TO HAVING A GOOD SAFETY PROGRAM. RETHINKING, YOUR ATTITUDE HELPS WITH FIRST IMPRESSION OF A NEW EMPLOYEE AND OFTEN HAS A LASTING EFFECT. EMPLOYEES YOU MEET FOR THE FIRST TIME APPEAR TO HAVE LITTLE RADAR SETS TUNED IN TO YOUR ATTITUDE. IF YOUR ATTITUDE IS POSITIVE, THEY RECEIVE A FRIENDLY, WARM SIGNAL AND THEY ARE ATTRACTED TO YOU. IF YOUR ATTITUDE IS NEGATIVE, THEY RECEIVE AN UNFRIENDLY SIGNAL AND TRY TO AVOID YOU.

A POSITIVE SUPERVISOR OR MANAGER CONTRIBUTES TO THE PRODUCTIVITY OF OTHERS. A NEGATIVE SUPERVISOR DOES NOT.

ATTITUDES ARE CAUGHT MORE THAN THEY ARE TAUGHT!

BOTH NEGATIVE AND POSITIVE ATTITUDES ARE TRANSMITTED ON THE JOB. THEY ARE PICKED UP BY OTHER EMPLOYEES.

A PERSISTENTLY NEGATIVE ATTITUDE, LIKE A ROTTEN APPLE IN A BARREL, CAN SPOIL THE POSITIVE ATTITUDES OF OTHERS. IT IS VERY DIFFICULT TO MAINTAIN A HIGH LEVEL OF PRODUCTIVITY AND SAFETY ATTITUDE WHILE WORKING NEXT TO OR WITH A PERSON WITH A NEGATIVE ATTITUDE.


AS YOU PROJECT YOUR OWN ATTITUDE ABOUT SAFETY TO THE EMPLOYEE'S, YOU CAN SEE THAT SAME ATTITUDE SPREAD AMONG THE WORKERS. IF YOU REACT INDIFFERENTLY, SO WILL EVERYONE ELSE. BE ALERT IN YOUR OWN SUPERVISOR SAFETY MEETINGS AND IMPLEMENT THE SAFETY PROGRAM WITH ENTHUSIASM.

INSTEAD OF LETTING YOUR WORKERS DEVELOP AN ATTITUDE OF "LOOK OUT, HERE COMES THE SAFETY INSPECTOR" - EDUCATE THEM TOWARD DEVELOPING THE MORE POSITIVE OF: "MY SUPERVISOR DOESN'T WANT ME TO TAKE CHANCES AND LETS ME KNOW ABOUT IT ANY TIME I BREAK A

SAFETY RULE."

REMEMBER, AS JAMES O'TOOLE SAID: "THE RIGHT KIND OF LEADERSHIP IS  
MORE LIKE TEACHING THAN COMMANDING."

YOUR FRIEND IN SAFETY,

 1997  
RAYMOND RUSH





*POSTER SESSION PAPERS*

# 46th Oilseed Conference

**Processing Efficiency:  
Meeting the Challenge**

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# POSTER ABSTRACT FORM

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**■ Poster Session:** All papers at this Conference are presented by invited speakers. The volunteer papers may be presented as posters. Your abstract will be reviewed by the organizing panel. Notification of acceptance will be made by December 16, 1996. The accepted poster papers will be published in the Conference Program. Due to space limitations, only 15 poster presentations will be accepted.

**■ Deadlines:** The abstract must be received by the Poster Chairperson by December 2, 1996. The final paper must be received by the Technical Chairperson by January 15, 1997. The final papers need to be processed, so early submissions by first-class mail or expedited delivery are strongly encouraged.

Mail your completed abstract form and final presentation paper to:

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#### Supercritical Fluid Extraction of Oil from Oilseed An AOCS/AOAC Collaborative Study

David L. Berner, Leslie J.D. Myer, and J.D. Colburn

The AOCS/AOAC recently completed a collaborative study on the supercritical fluid extraction (SFE) of oil from oilseeds. The study was conducted in 17 laboratories and 8 countries and included the soybean, cottonseed, rapeseed, safflower, and sunflower seeds.

Two SFE methods were studied. The first was designed to emulate AOCS method Ac 3-44 which is used to predict the refinable oil content of an oilseed. The second method emulates the FOSFA International method, Am 2-93, which is used to predict the total oil content of a seed. The first SFE method used 100% CO<sub>2</sub> as a solvent, while the second method used CO<sub>2</sub> modified with 15% ethanol.

Figure 1 illustrates the extraction kinetics using supercritical CO<sub>2</sub> as a solvent. A 30 minute SF extraction of ground soybeans yielded nearly identical results to AOCS method Ac 3-44. Increasing the extraction time to 45 minutes yielded oil recoveries equivalent to Am 2-93 (FOSFA) method. Subsequent experiments with other oilseeds showed that ethanol modified CO<sub>2</sub> was necessary for results equivalent to the FOSFA method. Figure 2 compares the results of 2 soybean and 2 sunflower samples for the four methods studied.

Statistical review showed that the SFE methods were equivalent to the corresponding AOCS methods. The AOCS has approved both SFE methods for the extraction of oil from oilseeds.



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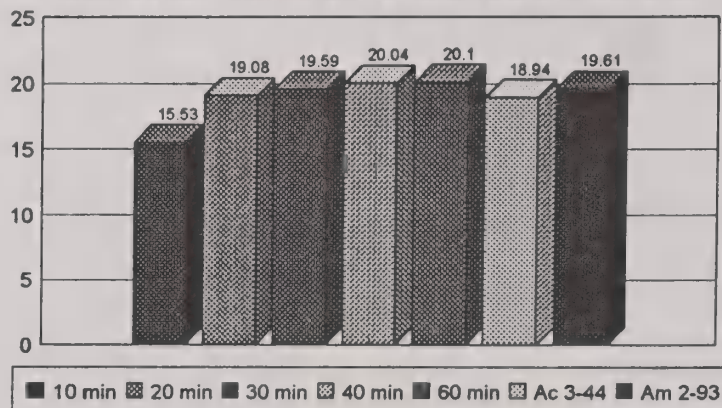
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Figure 1

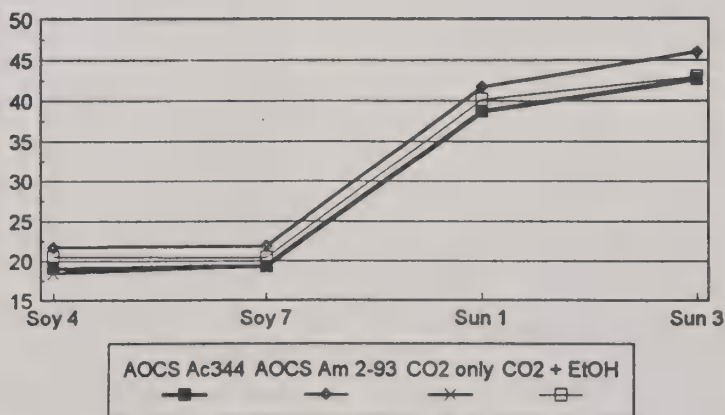
### SFE of Oil from Soybeans Extraction Kinetics vs Standard Methods



7500 psi, 100°C 2.0 mL/min min, 60 min

Figure 2

### SFE of Oil from Oilseeds Comparison of Methods



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# Characterization of soapstock from corn germ and peanut oil refining

Michael K. Dowd

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## Introduction

Soapstock is formed by the addition of sodium hydroxide to crude vegetable oil, and it is the main co-product of the oil refining process. In oilseed crushing operations, it is often added directly to the defatted meal to reduce dust, improve pelleting, and increase the energy content of the meal. In refining operations, it is typically acidified to recover fatty acids, which are sold as an animal feed supplement. Few other uses have been identified.

While soapstock is known to contain fatty acids, mono-, di-, and triglycerides, phospholipids, glycerol, and sterols, little detailed information on its composition is available. As part of an ongoing project to study and develop additional uses for soapstock, methods have been developed to characterize this material. This report describes the chemical and chromatographic analysis of soapstocks from the refining of corn and peanut crude oils.

## Material and Methods

AC Humko (Champaign, IL) and Lou Ana Foods (Opelousas, LA) donated soapstocks produced from corn germ and peanut crude oils, respectively. Moisture and residual solvent were determined by drying in a forced-draft oven at 105 °C for 24 hr. Phosphorus and sodium were measured by nitric acid digestion and inductively coupled plasma emission spectroscopy. Total fatty acids, neutral oil, and nitrogen were determined by AOCS methods G 3-53, G 5-40 and Ba 4e-93, respectively [1]. Values reported are the average of at least two determinations.

Trimethylsilyl derivatization was used to increase the volatility of soapstock components. Samples (~10 mg or ~100 mg) were derivatized with pyridine (2 ml), hexamethyldisilazane (2 ml), and trifluoroacetic acid (0.2 ml). Cholesterol-methyl ether (0.8 mg) was added as an internal standard. A Hewlett-Packard 5890 Series 2 plus gas chromatograph fitted with a 15 m J & W Scientific DB-5ht capillary column was used with helium as the carrier gas. The instrument was operated in constant flow mode (~1 ml/min) with a split injector (1:50) and flame ionization detector. Both injector and detector operated at 340 °C. The oven temperature profile was 100 °C for 3 min, 10 °C/min to 150 °C, 5 °C/min to 250 °C, 10 °C/min to 360 °C, which was held for 15 min. Both samples were analyzed in triplicate.

## Results and Discussion

The results of the chemical analyses are given in Table 1 and are compared with similar determinations for a series of cottonseed soapstocks [2]. Moisture and residual solvent was higher in the peanut sample than in the corn sample, but both values were within the range of

values reported for cottonseed soapstocks. Total fatty acids were similar in the corn and peanut samples, but neutral oil content was considerably greater in the peanut sample. The peanut sample contained double the sodium of the corn sample. Phosphorus and nitrogen were low in the peanut samples compared with the corn and cottonseed results. Because of the likely presence of phytic acid in the corn sample and the lack of information on the phospholipid distribution in peanut crude oil, phospholipids cannot be accurately estimated from the phosphorus values. The previously reported observation among cottonseed soapstock samples that higher levels of phosphate are associated with reduced total fatty acids is also apparent from the data.

Chromatography separated several soapstock components (Fig. 1). These are listed in Table 2 with their relative retention times and concentrations. Fatty acids included measurable levels of myristic, palmitic, stearic, oleic and linoleic acids. The distributions of the principal fatty acids in both soapstocks were similar to the fatty acid distribution of the individual oils. The peanut sample, though, did not contain concentrations of longer chain fatty acids in proportion to their reported content in oil [3]. Traces of several other fatty acids were also detected.

Six components eluted during the chromatography of both samples that have not been identified (Fig. 1). These peaks appear to be two set of compounds that are related to the three dominant fatty acids in the samples. The areas of these peaks tended to correlate with the sodium concentration of the sample. This was confirmed with a cottonseed sample that did not exhibit these peaks. These unknowns are believed to be degradation or rearrangement products that form in the high-temperature inlet in the presence of sodium and the trimethylsilyl-fatty acid esters. Decreasing the concentration of soapstock introduced into the split injector reduced the proportion of these compounds relative to the fatty acids. This approach was used to minimize the error in quantifying palmitic, oleic, and linoleic acids (chromatograms not shown).

Monoglycerides included 1-monopalmitin, 1-monooleitin, and 1-monolinolein and accounted for 1.1 and 0.58% (db) of the corn and peanut samples, respectively. 1-Monostearin, and 2-monopalmitin were also detected. Diglycerides with 34 and 36 acyl carbon atoms were present and accounted for 2.2% (db) of the corn samples and 2.8% (db) of the peanut sample. Significant levels of triglycerides were also detected. As noted before [2], the concentrations of soapstock triglycerides determined by chromatography were substantially less the levels of neutral oil determined by the AOCS method. In the peanut samples triglycerides accounted for 15.3% of the sample dry mass, while in the corn sample, they accounted for 11.0% of the dry mass.

The most concentrated sugar in both samples was sucrose. Raffinose was present in smaller concentrations. Polyalcohols identified included glycerol *myo*-inositol, mannitol, and sorbitol.

$\beta$ -Sitosterol, campesterol, and stigmasterol were measurable in both samples, and additional sterols were indicated. Glycosides containing all three sterol moieties were identified by mass spectroscopic analysis. These glycosides were found in the same relative concentrations as the free sterols. The carbohydrate component of these compounds could not be unambiguously identified by mass spectroscopy, but the principal peak among these compounds in both samples had retention times identical to the principal  $\beta$ -sitosterol-glycoside of cottonseed soapstock. This sterol-ether has been isolated, and the carbohydrate component identified as glucose [4]. The free sterol concentration was dramatically higher in the corn sample (4.9% db) than in either the peanut (0.81%) or cottonseed (average 1.3%) samples.

## Conclusions

Soapstocks from corn germ and peanut oil refining contain fatty acids, mono-, di-, and triglycerides, sterols, sterol-glycosides, carbohydrates, and polyalcohols. Relative to the average sample of cottonseed soapstock, corn and peanut soapstocks contained high concentrations of total fatty acids and low levels of phosphorus. The corn sample was characterized by having high levels of free sterols and of sterol-glycosides. The peanut sample contained triglycerides with twenty carbons and longer acyl chains, but the concentration of soap fatty acids with twenty carbon and longer acyl chains was quite small.

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Table 1. Chemical analysis of corn, peanut, and cottonseed soapstocks

Component <sup>a</sup>	Corn %	Peanut %	Cottonseed <sup>b</sup> (range, 39 samples) %
Moisture and residual solvent	37.9 (0.1) <sup>c</sup>	56.7 (0.2)	32.1 – 67.1
Total fatty acids	67.8 (0.6)	71.4 (1.1)	39.9 – 73.8
Neutral oil	23.9 (0.1)	33.5 (1.1)	5.6 – 55.7
Phosphorus	0.898 (0.018)	0.552 (0.009)	0.644 – 1.74
Sodium	4.56 (0.19)	9.86 (0.08)	3.4 ( $\pm 0.7$ ) <sup>d</sup>
Nitrogen	0.587 (0.009)	0.103 (0.002)	0.293 – 1.38

<sup>a</sup>Except for moisture and residual solvent all values are reported on a dry basis.

<sup>b</sup>Ref. 2.

<sup>c</sup>Parentheses indicate standard deviations.

<sup>d</sup>Average value calculated from fatty acid profile. Excess sodium from unreacted sodium hydroxide is not included. Analysis of a single representative sample yielded 3.9% sodium.



Table 2. Composition of corn, peanut, and cottonseed soapstocks by trimethylsilyl derivatization and gas chromatography

Component	Relative Retention Time <sup>a</sup>	Concentration, % (db) <sup>b</sup>		
		Corn	Peanut	Cottonseed (range, 39 samples)
<i>Phosphates</i>				
Phosphoric acid	0.124	tr <sup>c</sup>	0.177 (0.028)	
β-glycerophosphate	0.352	0.083 (0.029)	-	0.079 – 3.252
α-glycerophosphate	0.369	0.176 (0.018)	tr	0.025 – 0.954
<i>Fatty Acids</i>				
Myristic acid	0.395	0.031 (0.003)	0.066 (0.008)	0.101 – 0.409
Palmitoleic acid	0.488	tr	tr	0.021 – 0.387
Palmitic acid	0.504	8.62 (0.53)	4.89 (0.18)	4.14 – 15.1
Linoleic acid	0.596	17.82 (1.0)	7.48 (0.19)	8.41 – 25.2
Oleic acid	0.500	9.36 (0.28)	10.95 (0.36)	3.50 – 10.1
Stearic acid	0.613	0.507 (0.043)	0.458 (0.040)	0.447 – 1.39
Arachidic acid	0.727	0.076 (0.002)	tr	nd <sup>d</sup> – 0.131
<i>Monoglycerides</i>				
1-Monopalmitin	0.810	0.160 (0.008)	0.107 (0.018)	nd – 0.771
1-Monolinolein	0.891	0.277 (0.014)	0.211 (0.005)	nd – 2.054
1-Monoolein	0.894	0.653 (0.037)	0.267 (0.002)	nd – 0.730
<i>Diglycerides (by acyl carbon number)</i>				
D32		nd	nd	nd – 0.227
D34	1.289-1.295	0.440 (0.013)	0.805 (0.123)	nd – 2.33
D36	1.320-1.327	1.81 (0.07)	2.01 (0.23)	nd – 3.21
<i>Triglycerides (by acyl carbon number)</i>				
T48	1.507	0.312 (0.018)	0.608 (0.044)	nd – 0.388
T50	1.553	3.02	4.64	nd – 3.63
T52	1.610-1.623	6.80	6.00	nd – 9.51
T54	1.654	0.297 (0.040)	0.220 (0.064)	nd – 8.60
T56	1.681-1.727	0.655 (0.065)	1.44	nd – 0.547
T58	1.778	nd	2.595 (0.542)	nd – 0.874
T60	1.909	nd	tr	nd

Table 2 cont.

*Sterols*

Campesterol	1.088	1.27 (0.02)	0.126 (.004)	nd – 0.215
Stigmasterol	1.098	0.290 (0.004)	0.117 (0.005)	nd – 0.211
$\beta$ -Sitosterol	1.118	3.304 (0.048)	0.563 (0.019)	nd – 2.80
Campesterol-glycoside	1.370	0.414 (0.049)	0.057 (0.012)	tr
Stigmasterol-glycoside	1.373	0.127 (0.022)	0.050 (0.024)	tr
$\beta$ -Sitosterol-glycoside	1.384	1.34 (0.15)	0.361 (0.057)	0.889 – 2.84

*Polyalcohol and Carbohydrates*

Glycerol	0.127	1.014 (0.083)	0.078 (0.015)	0.310 – 5.06
Mannitol	0.460	0.069 (0.009)	0.032 (0.006)	tr
Sorbitol	0.464	0.037 (0.004)	0.039 (0.009)	tr
<i>myo</i> -Inositol	0.547	0.162 (0.068)	0.026 (0.006)	0.143 – 0.472
Sucrose	0.867	0.538 (0.052)	0.358 (0.047)	0.027 – 0.503
Raffinose	1.173	0.106 (0.017)	tr	0.025 – 1.069
Stachyose		-	-	nd – 0.163

---

<sup>a</sup>Retention times are relative to the elution time of methyl-cholesterol ether.

<sup>b</sup>Parenthesis indicate standard deviation.

<sup>c</sup>tr = trace.

<sup>d</sup>nd = not detected.

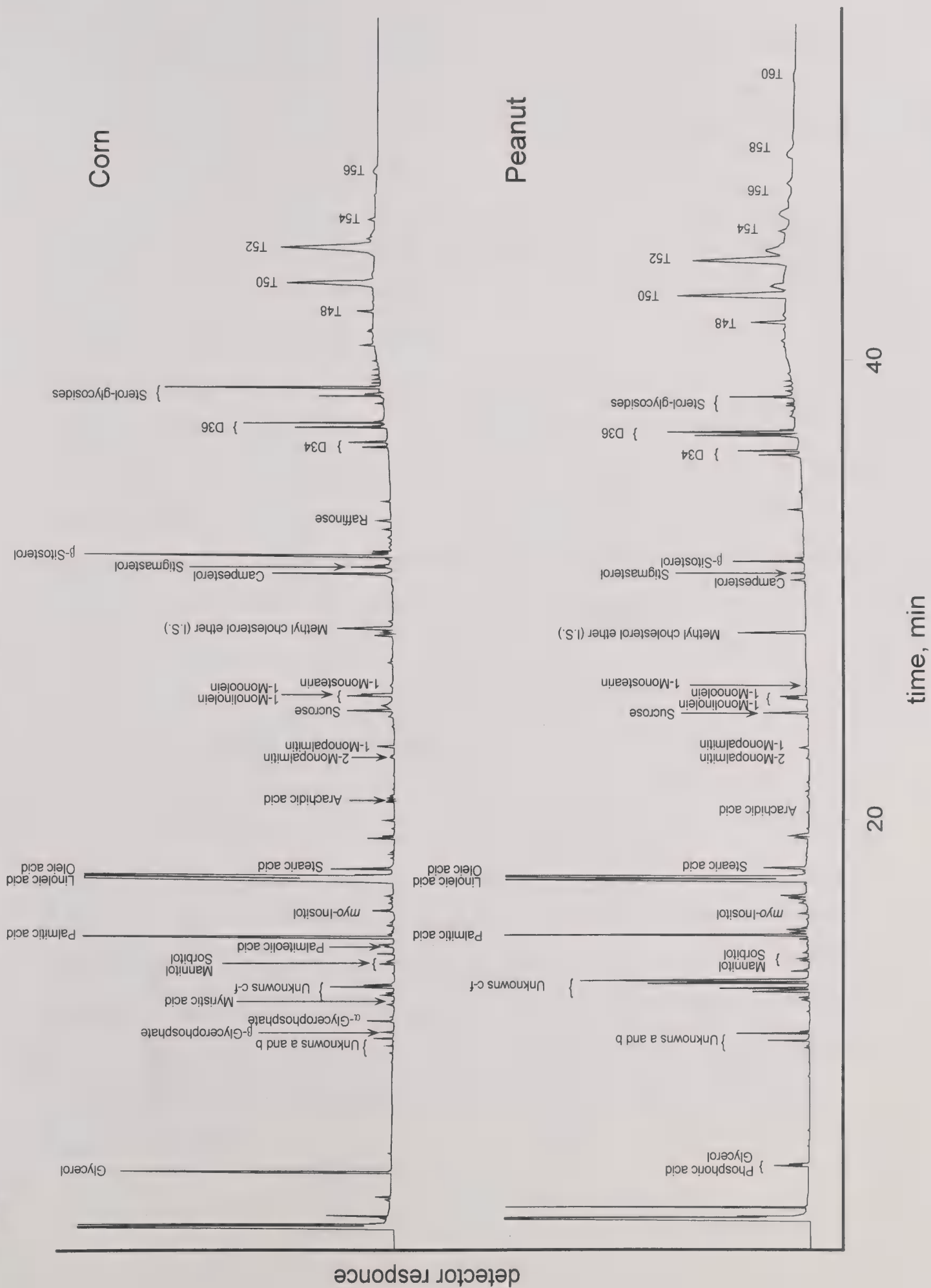


Fig. 1. Chromatogram of trimethylsilyl derivatized corn and peanut soapstocks.





## CHARACTERIZATION OF ELEOSTEARIC ACID PRODUCTION IN TUNG NUT HOMOGENATES

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Eleostearic acid is a conjugated trienoic fatty acid that accumulates to very high levels in the storage oils of tung nuts. The reactive nature of the conjugated bond system promotes the polymerization of tung oil upon exposure to air and heat. This property has made the oil a valuable commodity to the paints and coatings industries, which import large amounts of tung oil from South America and China. However, the supply of tung oil has been somewhat erratic, which has resulted in fluctuations of price and availability. Other vegetable oils such as soybean oil pose a different kind of problem to farmers. Soybeans are generally utilized for both their rich protein meal and high content of oil. However, increased dietary awareness and other economic factors have led to a general decrease in the demand for soybean oil. As a result, there is an excess of soybean oil that cannot be sold by farmers and other processors of soybeans. The goal of our research is to address the economic problems of both tung and soybean oil through a single objective. That is, to clone the genes that are responsible for the synthesis of eleostearic acid and use these genes to convert surplus vegetable oils into tung-like "drying oils." Success in this area will provide alternative uses for the excess oils and provide a new, reliable source of eleostearic acid for the relevant industries.

Our basic experimental design was to first develop a simple assay to characterize eleostearic acid production in a crude tung nut homogenate and then fractionate the homogenate to purify the proteins that catalyze the reaction. This is traditionally viewed as a reverse genetic approach, where the proteins of interest are first identified and then the corresponding genes are cloned. Our initial assay relied on the spectral properties of the eleostearic acid conjugated bond system, which has a characteristic absorbance pattern in the UV range. An increase in the absorbance pattern is a simple assay for an increase in eleostearic acid.

Tung nuts were harvested during the growing season from Blake Hanson's American Tung Oil orchard in Lumberton, Mississippi. Seeds were promptly removed and stored at  $-80^{\circ}\text{C}$  prior to usage. Verification that eleostearic acid biosynthesis was retained after frozen storage was demonstrated by incubating radiolabeled linoleic acid with tissue slices and identifying radiolabeled eleostearic acid by thin layer chromatography. After a lag phase of about 15 min, radiolabeled eleostearic acid increased linearly for about 30 min. To develop the spectral assay to measure eleostearic acid *in vitro*, seeds collected during the peak of oil synthesis were homogenized in a blender and centrifuged at  $20,000\times g$  to remove the excess oil by floatation. The remaining supernatant and pellet were reconstituted to generate a "cleared" homogenate. The homogenate was incubated with substrates and cofactors, aliquots were removed over time and extracted with hexane or chloroform, and the UV absorbance pattern of eleostearic acid was measured. Oleate, linoleate, and linolenate (or their acyl-CoA derivatives) could be used as substrates to produce eleostearic acid at an optimal rate of 4 nmol/min-mg protein. The reaction was linear for about 30 minutes, which was similar to the kinetics observed by radiolabeling experiments. No activity was observed in the absence of substrate or if the homogenate was boiled prior to the addition of substrate. Testing the activity in the  $20\times g$  supernatant and pellet alone demonstrated that the activity was clearly in the  $20\times g$  supernatant. Further fractionation of the  $20\times g$  supernatant at  $100,000\times g$  demonstrated that the activity resided in the  $100\times g$  supernatant. One difference between the activity in the  $20\times g$  and  $100\times g$  supernatant was that the  $100\times g$  supernatant required the acyl-CoA form of the substrate. No activity was detected using the free fatty acid.

Several experiments were performed to characterize the activity in the  $100\times g$  supernatant. The first objective was to determine what exogenous cofactors were required for the activity. In a typical reaction we added bovine serum albumin (BSA),  $\text{MgCl}_2$ , ferredoxin, NADH, flavin nucleotide reductase, and catalase in addition to the substrate. To test the importance of these factors, the  $100\times g$  supernatant was initially dialyzed using a 6-8 kDa dialysis membrane to remove endogenous small molecules. The exogenous cofactors were then left out one by one, or added back singly to reactions to test for their necessity. The results demonstrated that only  $\text{MgCl}_2$  and BSA had any significant effect on enzyme

activity. In addition, if the tung homogenate and subsequent 100Kxg supernatant were prepared in the presence of EDTA prior to dialysis, then Mg was not required. This suggested that an ion that might be displaced by Mg or chelated by EDTA could inhibit the production of eleostearic acid in the assay. To test for other inhibitors that might affect the production of eleostearic acid, several known oxygenase inhibitors were incubated with the 100Kxg supernatant prior to the reaction. Oxygenase enzymes are known to be involved in the desaturation of fatty acids and might be involved in the synthesis of eleostearic acid. Three classes of oxygenase inhibitors were tested: lipoxygenase inhibitors (NDGA, phenylpyridozolidinone, and salicylhydroxamic acid), epoxygenase inhibitors (clotrimazole and SKF) and a cyclooxygenase inhibitor (indomethacin). The only inhibitor that had any appreciable effect was the epoxygenase inhibitor SKF. The inhibition was dose responsive, but did not completely inhibit the activity. The optimal temperature for the enzyme assay was also investigated by testing temperatures ranging from 20 to 50 degrees in 5 degree increments. The results demonstrated that optimal activity was at 45 °C.

The results described above suggested that the enzymes for the production of eleostearic acid reside in the 100Kxg supernatant, and that an epoxygenase activity might be involved in the biosynthetic pathway. However, several additional results complicate this simple interpretation of the data. Experiments described below suggest that another activity, rather than true *de novo* synthesis, might contribute significant amounts of extractable eleostearic acid during the course of an enzyme assay. Furthermore, this eleostearic acid may come from a pre-existing pool that is not extractable by simple organic solvents at the beginning of an enzyme assay, but is converted to an extractable form during the course of the reaction. The strongest suggestion that there might be a pre-existing pool of inextractable eleostearic acid came from experiments in which stearoyl-, oleoyl-, linoleoyl-, and linolenoyl-CoA were all tested as substrates for the reaction in the 100Kxg supernatant. The results demonstrated that all of these acyl-CoAs promoted an increase of extractable eleostearic acid with approximately the same kinetics. It was therefore reasoned that a pre-existing pool of eleostearic acid might be converted to an organic extractable form in a time and substrate dependent fashion. Lipid transfer proteins were suspected to be involved in this process because these are small, soluble, abundant proteins that transport a variety of

lipids, including acyl-CoAs, between membranes within the cytosol. All of the experiments on the 100Kxg supernatant were done using chloroform extraction, which may not effectively dissociate lipids from proteins.

To test the possibility that eleostearic acid was associated with a protein component, the 100Kxg supernatant was incubated with increasing amounts of protease in the absence of substrate. The results demonstrated that the amount of chloroform-extractable eleostearic acid increased in direct proportion to amount of protease and length of incubation. The amount of eleostearic acid released was similar to the amount of eleostearic acid produced in a typical enzyme assay that included substrate. Also, the highest amount of eleostearic acid released by proteases was similar to the total amount of eleostearic acid present in the 100Kxg supernatant (determined by extensive extraction using a chloroform-methanol-salt procedure). These results demonstrated that eleostearic acid was indeed protected by a protein component and suggested that the acyl-CoA substrates might act to displace the lipids, either directly or indirectly, from the proteinaceous environment. It is reasonable to consider that lipid transfer proteins are involved in this process. The lipid transfer proteins could bind acyl-CoAs in the soluble fraction and deliver them to microsomal membranes enriched in eleostearic acid. An exchange reaction could take place that would allow acyl-CoAs to be incorporated into microsomes for further production of triglycerides while removing eleostearic acid-containing lipids for transport to the cytosol and deposition within developing oil bodies. Since the acyl-CoAs are added in excess the cycle would continue to transport eleostearic acid out of microsomal membranes. The eleostearic acid released into the aqueous environment might be the fraction that is readily extractable with chloroform. Although the true physiological significance of the chloroform-extractable lipids is unknown, the possibility that it represents a measure of cytoplasmic oil droplet formation is exciting. Very little is known about oil body biogenesis or its relation to the economic value of oilseed crops. An *in vitro* system would be extremely useful to dissect the protein machinery involved in this process.



These studies demonstrate that the simple enzyme assay based on incubation of a tung homogenate with substrate, extraction with organic solvent, and measurement of eleostearic acid absorbance may in fact be measuring several activities that can contribute to the increase in eleostearic acid. It is not yet known if true *de novo* synthesis of eleostearic acid is occurring in the 100Kxg supernatant. This can only be resolved by using radiolabeled, acyl-CoA forms of the substrates and measurement of radiolabeled eleostearic acid production in the 100Kxg supernatant. It is possible that the 100Kxg supernatant contains both a bulk displacement activity involving pre-existing eleostearic acid and a true enzymatic activity for the synthesis of eleostearic acid. Further experiments are being conducted to resolve these issues.

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# IMPROVING OIL YIELDS AND CO-PRODUCTS VALUES BY SEQUENTIAL EXTRACTION PROCESSING OF CORN

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## ABSTRACT

The Sequential Extraction Process (SEP) is a new approach to corn milling that applies principles commonly used in commercial soybean oil production and in soybean protein isolate production. SEP uses ethanol to first extract oil from flaked, dried, undegermed corn while simultaneously drying the alcohol (to 1% moisture) in a countercurrent system, and then extracts the protein by using a mixture of ethanol and dilute alkali. SEP recovers 95% of the oil in corn, which is superior to the 72% estimated recovery for conventional, prepress, hexane extraction of corn germ. Unfortunately, SEP oil has lower levels of triglycerides and tocopherols but higher amounts of free fatty acids, diglycerides, phospholipids and carotenoids than does hexane-extracted oil from corn germ. Zein, an important industrial protein, is recovered during SEP. A water-soluble, food-grade, glutelin-rich protein concentrate, possessing good functional properties, is also recovered. Hemicellulose extracted from SEP crude fiber has gum-like properties and can be used as stabilizer or thickener. The crude fiber and starch serve as substrates for fermentation into ethanol, thus increasing ethanol yield. These results demonstrate the potential of SEP to improve milling and ethanol production efficiencies and to produce new co-products with higher values than are achieved by ethanol plants employing wet or dry milling.

## INTRODUCTION

A substantial amount (19%) of the 7.4 million bushels of domestic corn crop produced in 1995 was processed by wet milling (1). The process involves an initial water soak to soften the kernels, followed by separation of the components by screening, centrifuging, and washing to produce starch, germ, and feed co-products such as corn gluten meal and corn gluten feed. Corn starch is used in the manufacture of sweeteners and for fermenting into industrial chemicals such as ethanol, isopropanol and acetone. Crude corn oil is obtained from the dried germ by using a combination of mechanical expression and solvent extraction, which recovers about 72% of the oil from corn (80% of the oil in corn found in the germ x 90% extraction efficiency). Corn gluten meal is a high-protein (60%) product of wet milling, but it is utilized only as feed because the sulfur dioxide used during steeping adversely affects the functional and edible properties of the protein and produces sulfites, which are potentially hazardous to human health. Wet milling recovers starch in greater yield and purity than does dry milling, and the co-products have commanded higher returns as feed ingredients; but, current wet-milling methods also use considerable amounts of capital and energy. In addition, traditional feed markets are becoming saturated with the co-products from wet corn mills, especially corn gluten feed (for cattle) whose traditional European market could become fixed as a result of trade negotiations. If there is to be continued growth in ethanol production in the United States, which many project as a result of the 1990 Clean Air Act, a new mix of co-products will be required to maintain and increase revenues. Co-product revenue can provide as much as 40% of the income in wet-milling/ethanol production.

The Sequential Extraction Process (SEP) (Fig. 1) has recently been developed as an alternative technology for fractionating corn (2, 3) and integrates well with ethanol production (but not with food-grade starch or corn sweetener production) to make the facility more cost effective by achieving low-cost ethanol drying and by recovering co-products with higher values. The basic elements of the process are: (i) the countercurrent percolation extraction of crude oil using ethanol, which can be produced from cornstarch fermentation; (ii) the simultaneous dehydration of ethanol during oil extraction through adsorption of moisture by dry corn solids, thus reducing the costs of drying the alcohol (4, 5); (iii) use of aqueous ethanol and ethanol mixed with dilute alkali to extract and recover zein and food-grade protein concentrate, respectively (6); and, (iv) recycling the ethanol produced from fermentation to upstream steps of extraction. The economic-engineering assessment of SEP showed that this process is economically feasible, that estimates for return on investment (ROI) are attractive under many likely market situations, and that several additional opportunities exist to further improve profitability (7). We have recently augmented SEP with new elements of hemicellulose recovery and converting the cellulose to ethanol to improve ethanol production and further add to the value of SEP co-products. This paper presents the results of our studies that determined the technical feasibility of SEP and evaluated the chemical and/or functional properties of its co-products.

## EXPERIMENTAL PROCEDURES

### Preparation of Corn

Twenty-five batches of soft dent corn (Pioneer 3377, Pioneer Hi-Bred International, Inc., Johnston, IA), each weighing 350 g, were prepared. Each batch was cracked and then flaked to 0.5 mm by using a Roskamp roller mill (Model K, Roskamp Mfg., Inc., Waterloo, IA). The flaked corn samples were dried at 50°C in a forced-air convection oven to a moisture content of 1.12%. Each dried sample was stored in a polyethylene bag and kept in a desiccator at ambient temperature until used.

### Proximate Analyses

All batches of corn were analyzed for crude free fat and crude protein (N x 6.25) by using AACC standard methods 30-20 and 46-08 (8), respectively. Moisture contents (MC) were determined by Karl Fischer titration (9). These methods were also used to analyze the fractions produced by SEP of corn.

### Extraction System

Countercurrent oil extraction was simulated by using a system that was adapted from percolation-type extractors commonly used by the soybean oil industry (Fig. 2). Jacketed glass vessels contained the dried, flaked, undegermed corn and the aqueous ethanol with concentrations ranging from 96.3% (weight basis (wt b), vessel 7) to 99.2% (vessel 1). The system was maintained at 56°C by circulating hot water through the vessels. Cold water condensers minimized solvent evaporation. Entry of atmospheric moisture was prevented by drying tubes and by flushing nitrogen gas throughout the system.

### Sequential Extraction Processing of Corn

SEP was performed according to the procedure of Hojilla-Evangelista et al. (2, 3). SEP used seven ethanol concentrations at start-up of the countercurrent extraction - 96.3% ((wt b), newest solvent), 97.8%, 98.5%, 98.8%, 99.0%, 99.1% and 99.2% (oldest solvent). Dried,



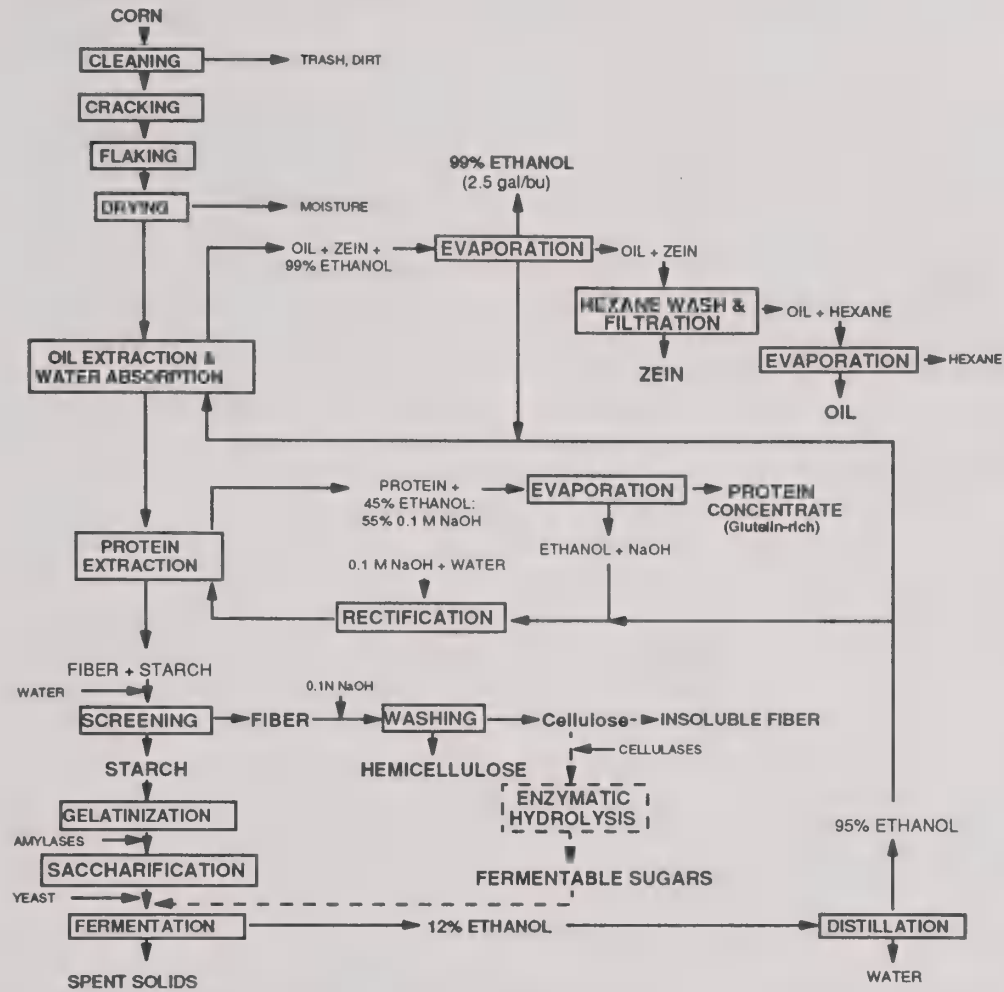


Fig. 1. Sequential Extraction Processing of Corn  
(- - - denotes optional steps)

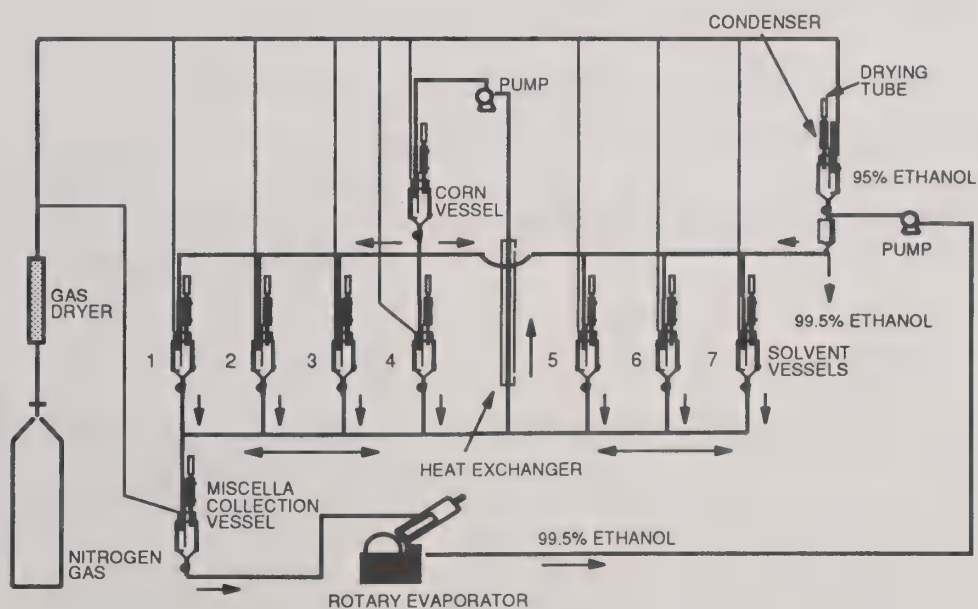


Fig. 2. Countercurrent Oil/Moisture Extraction System

flaked, undegermed corn was placed in the extraction vessel and was subjected to seven extraction stages by using a 2:1 ratio of solvent-to-corn. Oil extraction began with the solvent in vessel 1, which was pumped through the heat exchanger, allowed to percolate through the bed of flaked corn at a rate of 25 g solvent/min, then drained for 10 min into the previously-emptied vessel; i.e., solvent 1 is drained into the miscella recovery vessel, solvent 2 into vessel 1, solvent 3 into vessel 2, and so on until solvent 7. This series of steps advanced the miscella and simulated countercurrent solvent flow.

After the oil extraction and simultaneous ethanol dehydration step, the protein was extracted from the marc (solvent-laden, defatted, flaked corn) by using 45% ethanol-55% 0.1M NaOH as described by Hojilla-Evangelista et al. (2, 3). The starch and crude fiber remaining after centrifugation were separated according to the procedure adapted from our laboratory wet-milling method (10), which involved repeated suspension of the fiber/starch residue in water to form a slurry and filtration of slurry through 100- and 200-mesh sieves of the Ro-Tap testing sieve shaker (W.S. Tyler, Inc., Mentor, OH).

### **Evaluation of SEP Oil Quality**

Oil recovery was calculated by determining the difference between the crude free fat content of the unextracted flaked corn and the defatted flaked corn. For oil quality analyses, SEP crude corn oil was recovered by first evaporating the ethanol from the miscella using a rotary evaporator. The oil was separated from any proteinaceous solids by washing with hexane, filtering the mixture, and then evaporating the hexane by using a rotary evaporator. SEP corn oil and hexane-extracted corn oil (control) were analyzed for fatty acid profile, free fatty acid content, phosphorus content, color, carotenoid content, tocopherols content, and quantities of mono-, di-, and triglycerides (11).

### **Characterization of Corn Protein Concentrate**

Protein fractions extracted during SEP were identified by sodium dodecyl sulfate-polyacrylamide gel electrophoresis (SDS-PAGE) (12) and by amino acid analysis. The functional property tests performed on the SEP corn protein concentrates included solubility, foaming capacity and stability, heat coagulability (13), emulsifying capacity (14) and emulsion stability (15). These tests were also performed on freeze-dried soy protein concentrate prepared in our laboratory (defatted, acid-washed, low-temperature) for comparison purposes.

### **Recovery and Evaluation of Hemicellulose from SEP Crude Fiber**

The procedure for hemicellulose extraction was adapted from the methods of Rutenberg and Herbst (16) and Wolf et al. (17). The air-dried hemicellulose fractions were evaluated for color, dispersibility in water, viscosity, and heat stability (18). These properties were compared with those of gum arabic FT (TIC Gums, Inc., Belcamp, MD), which served as the control gum.

### **Fermentation of SEP Fiber and Starch to Increase Ethanol Yield**

Batch fermentations of air-dried fiber/starch residues were performed in Bioflo 3000 Bioreactors (New Brunswick Scientific, Edison, NJ) as described by Hojilla-Evangelista et al. (18). The amounts of solids, ethanol, and reducing sugars in the reactor broth was monitored by periodic sampling. Ethanol concentration was determined by using a Waters high-pressure liquid chromatograph (Millipore Corporation, Milford, MA) equipped with a Waters Model 401 refractive index detector, column heater, autosampler and computer controller. Reducing sugars were determined by the Somogyi-Nelson method (19).

## RESULTS AND DISCUSSION

### Oil Extraction with Ethanol

Ethanol was quite effective in extracting oil (Table 1), recovering nearly 95% of the total oil in corn and left less than 0.5% residual. This is more oil than is recovered by extracting germs, demonstrating the excellent oil extraction efficiency of ethanol by the countercurrent method used in SEP. However, SEP oil is darker in color than hexane-extracted oil, which we attribute to high xanthophyll content. This assumption was supported by Feng's (11) study, which determined that SEP oil contained ten times as much carotenoid pigments as hexane-extracted oil from corn germ (Table 2). Xanthophylls are more polar than carotenes and would be extracted more readily by the polar solvent ethanol than hexane, thus imparting darker color to SEP oil.

Unfortunately, SEP oil had much higher levels of free fatty acids than did hexane-extracted corn germ oil (Table 2), which may be explained by the extractability of free fatty acids by the more polar solvent ethanol and by the inclusion of corn endosperm (which contains 95% of the free fatty acids in corn kernels) for oil extraction (11). SEP oil also contained less amounts of triglycerides and tocopherols, but higher amounts of diglycerides, phosphatides, and waxes than did hexane-extracted oil (Table 2). The high levels of free fatty acids and waxes in SEP oil indicate that refining loss would be quite high.

### Ethanol Dehydration

The moisture content of the corn increased significantly during oil extraction (Table 1), indicating the simultaneous adsorption of water from the solvent by the flaked corn. The moisture content of the ethanol recovered from the evaporation of full miscella was reduced from an initial amount of 5% to 1.7% (volume basis, vb) (95% ethanol to 98.3% ethanol), which further verified ethanol dehydration during the oil extraction process. This moisture content is still higher than the industry's standard of 0.5%, and we are currently evaluating other factors to further reduce the moisture content of the ethanol from SEP.

### Protein Extraction

The non-oil solids co-extracted with the crude corn oil (Table 1) contained 25-30% protein, accounting for about 10% of the protein initially present in corn. This behavior was not unexpected because ethanol is capable of solubilizing and extracting small amounts of protein during oil extraction. These non-oil solids were identified by SDS-PAGE and amino acid analysis as predominantly zein, which has high-value applications in industrial products.

Nearly two-thirds of the total protein was extracted by the ethanol-alkali from soft dent corn (Table 1), a recovery higher than that observed by Chen and Hoff (20) when a mixture of 50% ethanol and 0.08M NaOH was used to extract protein from ground corn. The freeze-dried corn protein extracts contained more than 80% crude protein (db, Table 1), which was significantly greater than the typical 60-62% protein content of corn gluten meal. SEP protein concentrate was white, had a mild corn flavor, and may be considered food grade because all chemicals used in SEP are allowed for processing food. Its light color (compared with the bright yellow color of corn gluten meal) is desirable for food applications because little added color will be imparted to the product.

SDS-PAGE results indicated the presence of glutelins, zein and albumins in the freeze-dried SEP protein concentrate (3). The amino acid profile of the SEP protein concentrate showed that the amounts of essential amino acids in the concentrates did not differ markedly from



Table 1. Recoveries and Compositions of Products from Soft Dent Corn Achieved by SEP

<b>Oil Extraction</b>	
Initial crude fat in flaked corn (% dry basis, db)	3.4
Residual oil (% db)	0.2
Oil recovery (%)	94.7
<b>Ethanol Drying</b>	
Initial MC in flaked corn (%)	1.1
Corn MC after oil extraction (%)	3.3
Initial ethanol MC (% vb)	5.0
Ethanol MC after extraction (% vb)	1.7
<b>Crude Protein (CP) Extraction</b>	
Initial CP in flaked corn (% db)	8.3
Zein extracted with oil	
Percent of total CP with oil ( <b>ZeIn</b> ) (db)	11.6
Protein concentrate from ethanol:NaOH extract	
Percent of total CP extracted (db)	66.1
CP in freeze-dried extract (% db)	83.1
Residual crude protein in fiber/starch	
Residual CP (% db)	1.7
Unrecovered CP (%)	21.8

Table 2. Contents of Various Lipid Classes in Hexane-extracted and SEP Corn Oil

Lipid class	Hexane-extracted oil from corn germ	SEP oil from whole corn
Free fatty acids (%)	1.9	9.4
Triglycerides (%)	92.6	63.8
Monoglycerides (%)	trace	trace
Diglycerides (%)	2.2	3.5
Phosphatides (%)	0.24	1.74
Carotenoids (%)	0.0006	0.0054
Tocols (%)	0.046	0.027
Waxes (%)	3.0	21.5

those found in their respective whole-corn values, but were much improved over those of corn gluten meal. A similar trend was observed for the nonessential amino acids. These results suggest that SEP was not detrimental to the corn amino acids, especially to lysine, the limiting amino acid in corn and other cereal crops.

### Some Functional Properties of SEP Corn Protein Concentrate

**Solubility.** The SEP corn protein concentrate was markedly more soluble than the freeze-dried soy protein concentrate at pH values > 3. More than 80% of the corn protein remained soluble in water at pH values of  $\geq 7$  (Fig. 3). This behavior appears to confirm the SDS-PAGE results that showed the presence of glutelins and albumins in significant quantities. Through SEP, we have recovered a corn protein concentrate that is highly-soluble within a pH range found in most food systems.



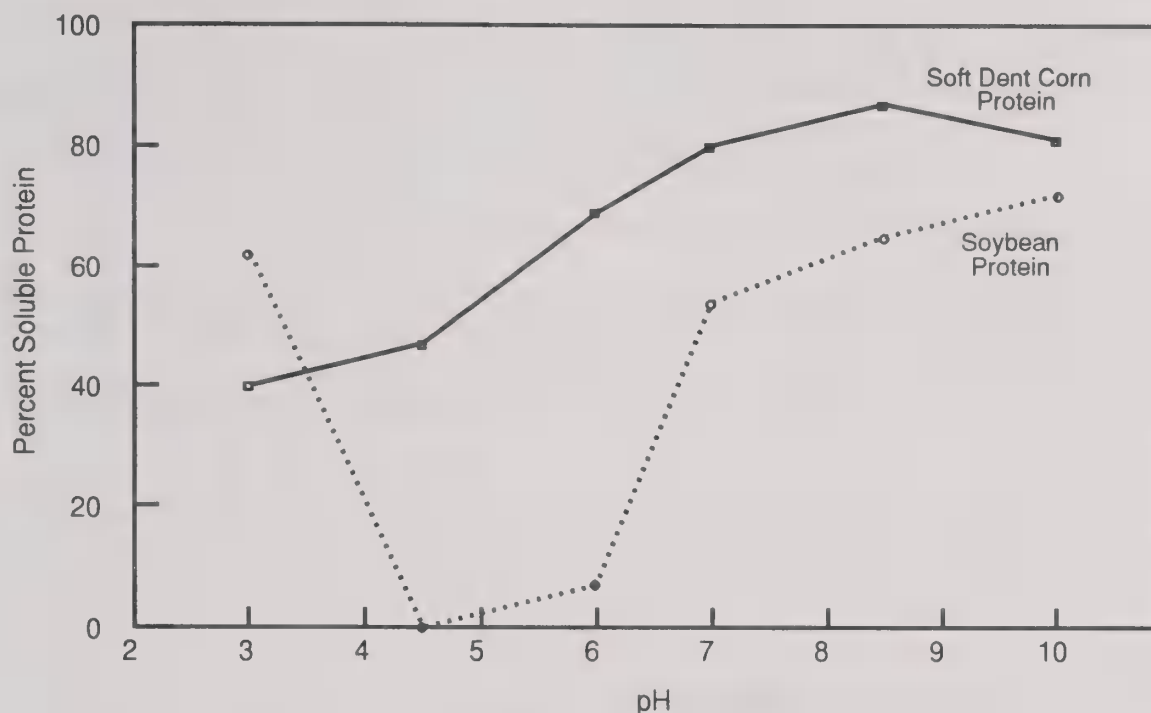


Fig. 3. Solubility Profiles of Soy and SEP Corn Proteins

Table 3. Some Functional Properties of Soy and SEP Corn Protein Concentrate

Functional property	Protein concentration	Soft dent corn	Soybean
Emulsifying capacity (g oil/g protein)		869	639
Foaming capacity (mL)	0.1%	145	53
	1.0%	102	147
Foam stability (% foam left after 15 min)	0.1%	2	10
	1.0%	29	98
Heat coagulability at 100°C (% loss in solubility)		5	36

**Emulsifying Properties.** Emulsifying capacity measures the amount of oil that can be emulsified by protein, before collapse of the emulsion occurs. The ability of protein to bind fat is important for such applications as meat extenders and replacers because it enhances flavor retention (21). SEP protein concentrate had significantly greater emulsifying capacity and better stability than the protein from soybean (Table 3).

**Foaming Properties.** Foaming is an important functionality of proteins in such products as cakes, whipped toppings, and icings. Dilute (0.1%) solutions of SEP corn protein concentrate produced foam volumes that were significantly greater than that produced by the same concentration of soy protein concentrate (Table 3). Foam volumes of 0.1% corn protein solutions were nearly equal to that produced by 1% soy protein concentrate, but they were very unstable and readily collapsed (Table 3). Increasing the protein concentration to 1% improved foam stability of the soy protein substantially, but only marginally for the SEP corn proteins.

**Heat Coagulability.** Heat coagulability of the protein was expressed as the percent loss in solubility after heating 1% protein solutions at 100°C for 20 min. The SEP corn protein concentrates demonstrated greater heat stabilities than did the soy protein concentrate (Table 3). This property implies that, unlike soy protein, SEP corn protein concentrates may be used in many high-temperature, short-time processes without losing their functionality.

### **Extraction of Starch, Crude Fiber, and Hemicellulose**

The fiber/starch separation method gave a crude fiber yield of 14.4% (db) and a starch yield of 67.8% (db) based on the weight of air-dried, defatted, flaked corn prior to protein extraction. The residual protein remained soluble in the water-wash (confirmed qualitatively by the Biuret test), which indicated that the predominant protein classes were albumins.

The alkali used in the protein extraction step of SEP is believed to have a solubilizing effect on the fiber fraction, thus making the hemicellulose readily available for subsequent extractions. The recovered hemicellulose could increase the co-product value of corn fiber because of its gum-like properties. The air-dried SEP hemicellulose produced from pooled extracts 1 and 2 was a white, fine powder, while that recovered from pooled extracts 3 and 4 was grayish-white, porous, and granular. Other workers (16, 17, 22) reported that the color of corn hull hemicellulose powder ranged from white to tan. The average yield of hemicellulose from all extracts was 42.6% (18), which compared favorably with values of Wolf et al. (17), who reported that 32-40% of hemicellulosic material could be obtained from coarse fiber from wet-milled corn, while higher yields (41-46%) could be recovered from laboratory-prepared pericarp and bran from dry-milled corn.

### **Some Properties of Hemicellulose**

**Dispersibility in water.** The SEP hemicellulose powder from extracts 1 and 2 was dispersible in water at room temperature but required a longer stirring time (5 min) than did gum arabic (less than 1 min) to completely disperse. The hemicellulose from extracts 3 and 4 did not completely disperse even after 15 min stirring. This contrast in dispersibilities of the two sets of hemicellulose extracts is indicative of compositional differences, which warrants further study. Dispersion in water may be improved by recovering the SEP hemicellulose in spray-dried and agglomerated form.

**Viscosity.** SEP hemicellulose solutions, specifically pooled extracts 1 and 2, were significantly more viscous than gum arabic solutions of the same concentration, indicating that less SEP hemicellulose would be needed to provide the same viscosity of a given solution of gum arabic. The viscosity of pooled extracts 3 and 4 was only 20% of the viscosity of extracts 1 and 2, but still higher than that of gum arabic (Fig. 4). The viscosity of gum arabic remained unchanged over the pH range studied. For the hemicellulose samples, the highest viscosity readings were recorded at the "normal" pH of 4.3-4.5. Viscosity of extracts 1 and 2 gradually declined as pH increased, but viscosities of other hemicellulose samples generally remained constant over the pH range of 5.5-7.5.

The resulting viscosities of hemicellulose solutions, however, were still relatively low for the concentration tested (5%). This attribute of having high solids content but low viscosity provides adhesive properties to hemicellulose solutions that would be useful for candy coatings and breadings and batters in frozen foods.

**Heat stability.** There were no endothermal peaks observed for the SEP hemicellulose and gum arabic samples during DSC, which indicated good heat stability of the samples because there were no thermal changes when the samples were scanned from 30-150°C.

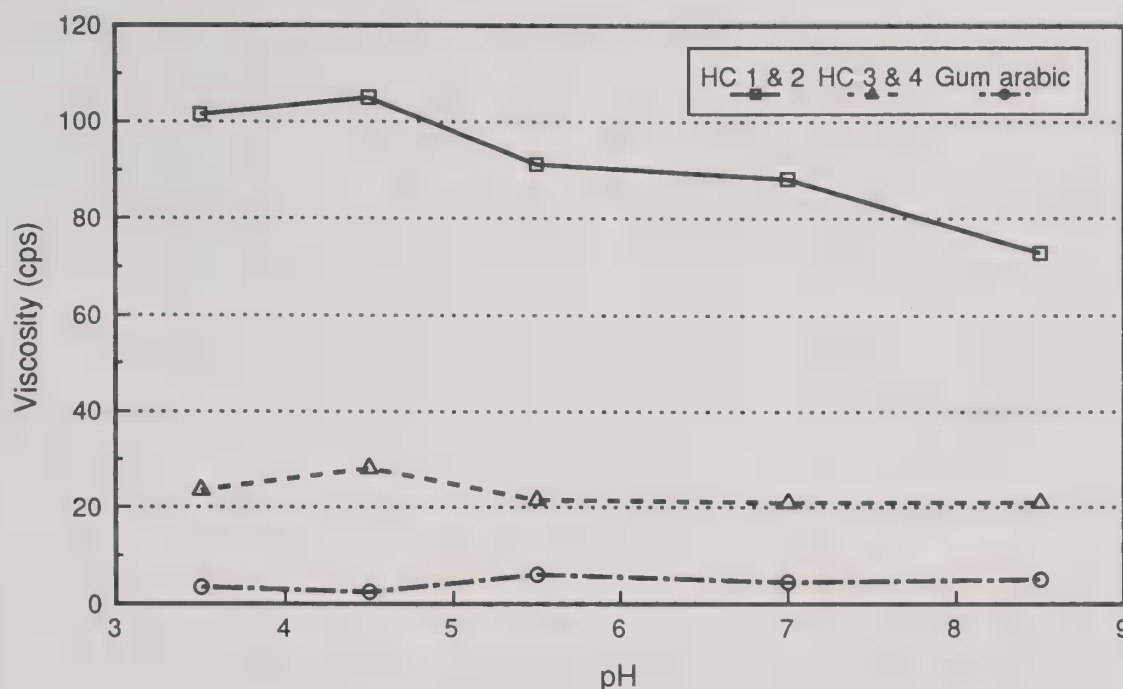


Fig. 4. Effect of pH on viscosity of hemicellulose (HC) and gum arabic

### Fermentation of SEP Starch and Crude Fiber

The maximum amount of ethanol produced from the fermentation of degermed soft dent corn was 19% more than that produced from the fermentation of undegermed corn (Table 4). The presence of oil apparently interfered with fermentation of the latter substrate and reduced not only the ethanol yield but also the rate of ethanol production.

The maximum ethanol yields from the degermed and undegermed dent corn substrates were both less than those obtained from the SEP starch/fiber substrates (Table 4). The maximum ethanol yield for SEP-1 (starch/fiber with hemicellulose) was 5% higher than that of degermed corn and 25% greater than that of undegermed corn. The ethanol yield for SEP-2 (starch/fiber without hemicellulose) was the highest amount among the four substrates. This ethanol yield (basis solids) was 28 and 52% more than those of degermed and undegermed corn, respectively. The differences in ethanol yields, however, were not very significant when the data were expressed in terms of gallons of ethanol produced per bushel of whole corn (Table 4). The presence of hemicellulose in SEP-1 had a negative effect on ethanol production, even though the fiber fraction had been partially delignified by contact with alkali during protein extraction. Extracting the hemicellulose fraction (SEP-2) increased ethanol yield by almost 15%, indicating that enzymes had easier access to the fiber for hydrolyzing into reducing sugars. This finding supported our assumption that the ethanol-alkali treatment during SEP can potentially delignify corn fiber when extracting the protein concentrate. Lignin is intimately associated with cellulose and hemicellulose and restricts access of hydrolytic enzymes to these polysaccharides. The reduction in lignin content during SEP will make cellulose and hemicellulose more accessible to hydrolytic enzymes, thus producing more fermentable sugars and, consequently, increasing ethanol yields.

The yields of spent solids from all substrates (Table 4) were considerably lower than the 17.5 lb/bu reported for distillers' dried grains. The hydrolysis of fiber by cellulases and the removal of some corn components, such as germ, starch, and lignin and hemicellulose from SEP fiber, reduced the amount of spent solids from fermentation as would be expected.



Table 4. Ethanol and Spent Solids Yields and Rate of Ethanol Production from Fermentation of Various Corn Substrates

Fermentation substrates	Maximum ethanol yield		Ethanol production g/L/hr	Spent solids yield	
	g/L substrate	gal/bu whole corn		g/100 g substrate	lb/bu whole corn
Undegermed corn	25.2	2.2	2.0	13.3	7.4
Degermed corn	30.0	2.3	2.6	13.1	6.5
SEP-1	31.6	2.1	1.6	9.1	3.2
SEP-2	38.4	2.4	2.0	6.5	2.6

### SUMMARY

The sequential extraction processing of dried, flaked, whole corn was a technically viable process for ethanol production and has potential to produce more valuable co-products than alternative processes (Table 5). Oil yields are maximized because the entire corn kernel is used for extraction. Oil is recovered on-site, eliminating the need to ship the germ to a separate crushing facility and minimizing deterioration in oil quality. Expensive ternary distillation for recovering anhydrous ethanol is replaced with a simple water-adsorption step using flaked dried corn as the adsorbent. Oil extraction and ethanol drying are accomplished in one step. All of the steps used in SEP are suitable for food, unlike in wet-milling where steeping corn in sulfur dioxide (SO<sub>2</sub>) solution relegates the protein to feed uses. Eliminating exposure of corn oil to SO<sub>2</sub> also will give the oil a premium because it is more stable to oxidative rancidity. In addition, the co-products from SEP (zein, food-grade glutelin-rich protein concentrate, hemicellulose) possess properties that could be utilized in both food and nonfood applications and do not add to the growing glut of protein and fiber feeds.

Table 5. Values of Co-products from Wet Milling and SEP

Product	Selling price <sup>1</sup>	Yield	Value (\$)
<b>WET MILLING</b>			
Corn oil	\$0.23/lb	0.6 lb/bu	0.37
Corn gluten feed	\$0.05/lb	14.0 lb/bu	0.68
Corn gluten meal	\$0.15/lb	3.4 lb/bu	0.50
Ethanol	\$1.22/gal	2.3 gal/bu	2.81
Total			<b>4.36</b>
<b>SEP</b>			
Corn oil	\$0.23/lb	1.8 lb/bu	0.41
Zein	\$2.40/lb <sup>2</sup>	0.5 lb/bu	0.20
Food-grade protein concentrate	\$1.10/lb <sup>2</sup>	3.0 lb/bu	3.30
Ethanol	\$1.22/gal	2.4 gal/bu	2.93
Hemicellulose	\$0.50/lb <sup>2</sup>	3.3 lb/bu	1.65
Spent solids	\$0.06/lb	2.9 lb/bu	0.17
Total			<b>8.66</b>

<sup>1</sup> From the Chemical Marketing Reporter (23), unless otherwise noted.

<sup>2</sup> Estimated likely prices.



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Estimation of Hildebrand's Solubility Parameter  
For Oilseed Lipids and Solvents

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## Abstract

In oilseed extraction selective lipid recovery is often desired, requiring design of a complex extraction agent. This requires evaluating Hildebrand's solubility parameters of solutes and the extracting agent. The values of H (Hildebrand's) parameter for oilseed triglycerides range between 6.5 to 7.5. Because hexanes/heptanes have the values of the solubility parameter between 6.5 and 7.5, these solvents have ideal solubility values for extracting oil. However, these solvents poorly extract polar lipids such as sterols and biphenyls. It was found that supercritical CO<sub>2</sub> could be used to estimate the necessary values for designing selective extraction agents for sterols, biphenyls and oils. Examination of oilseed lipid extraction data with supercritical CO<sub>2</sub> showed a relationship between the H parameters and extraction. A method utilizing the estimated values of the Hildebrand's solubility parameters to design a liquid extraction agent is presented.

## Hildebrand's Solubility Parameter

### Definition

The Hildebrand's solubility parameter (1) for a solute in a solvent is defined as follows.

$$\delta = (\Delta E/V^1)^{1/2} \quad (1)$$



where  $\delta$  denotes the solubility parameter,  $E$  the internal energy required for phase change and  $V^l$  the liquid molar volume, respectively. Equation (1) may be rewritten as below.

$$\delta = [ (\Delta H - RT) / V^l ]^{1/2} \quad (2)$$

where  $\Delta H$  represents the heat of vaporization at given  $P$  and  $T$  (pressure and temperature), and  $R$  for gas constant. Although there are other expressions using critical properties for predicting the Hildebrand's parameters for a single component, equation (2) is frequently preferred. The relationship between the solubility parameters and the energy of mixing required to have a solute solubilized in a solvent is expressed as follows.

$$\Delta E^M = f\{\phi_1, \phi_2, V_m^1\} * (\delta_1 - \delta_2)^2 \quad (3)$$

where  $\Delta E^M$  represents the energy required for the mixing of the two components (solvent and solute),  $f$  a function,  $\phi$  is molar volume fraction, subscripts 1, 2 and  $m$  for component 1 (solute), component 2 (solvent), and mixture, respectively. It may be seen from equation (3) that as the values of  $\delta_1$  and  $\delta_2$  approach each other,  $\Delta E^M$  is getting smaller. The smaller the  $E^M$  value becomes, the higher the solubility of component 1 is expected. The state of infinite miscibility would be attained when the values of solubility parameter of the solvent and solute become exactly equal.

#### Estimation of $\delta$

Regardless of oilseed species, the majority of oilseed lipids consist of triacylglycerol (TAG), with the number of acyl carbons varying between 42 and 60. With a few exceptions, this number falls between 48 and 54. The minor oilseed lipids include phosphorus compounds, sterols, phenolics, and traces of miscellaneous compounds (flavonoid, polysaccharide and others). Among these oilseed lipids, major TAG components and well-known plant sterols were selected to estimate their values of the solubility parameter, along with a few typical alternate solvents to hexane (3,4). Gossypol, a unique binaphthyl compound of cottonseed, was also selected for the estimation.

### Solvent

Major typical alternative solvents to hexane investigated to date by the oilseed industry include iso-hexane ( $i\text{-C}_6$ ), heptane ( $n\text{-C}_7$ ), ethyl alcohol (EtOH), iso-propyl alcohol (IPA), and supercritical carbon dioxide ( $\text{SC-CO}_2$ ) (3-5). To estimate the solubility parameter for these solvents except  $\text{SC-CO}_2$ , equation (2) may be used with  $\Delta H$  estimated at the expected extraction temperature. The extraction temperature is usually equal to NBP or slightly lower. With the estimated values for the latent heat and the liquid molar volume, based upon the liquid density at the extraction temperature, the values of the solubility parameter were computed and given in Table 1. Methanol (MeOH) and pentane

(n-C<sub>5</sub>) were included in Table 1 to show the pattern of  $\delta$  for alkanes and alcohols.

The following equation suggested by Giddings (2) has been used for estimating  $\delta$  of SC-CO<sub>2</sub>.

$$\delta_{sc} = (\rho_{liq} / \rho_{sc}) * 1.25 * P_c^{1/2} \quad (4)$$

where  $\delta$ , the Hildebrand's solubility parameter, is in the unit of (cal/cc)<sup>1/2</sup>,  $P_c$  critical pressure in the unit of (atm),  $\rho$  denotes density, subscripts "liq" liquid, and "sc" supercritical phase, respectively. As noted from Equation (4),  $\delta$  of SC-CO<sub>2</sub> is dependent upon  $\rho$  of SC-CO<sub>2</sub>. The solubility parameter of a typical liquid extracting agent is only dependent upon temperature, while that of a supercritical fluid is a function of both pressure and temperature.

#### Oilseed Lipid

The  $\delta$  of TAG, sterols and gossypol may be estimated using equation (2), in which  $\Delta H$  information is required. Because of the extremely low vapor pressure, the heat of vaporization for the oilseed lipids has rarely been investigated and scarcely reported. Devoid of the required data, one has to utilize the established thermodynamic equations to predict the data. Two types of well-known equations are available for the prediction. One is the Claussius-Clapeyron type equation (1), utilizing vapor pressure data, and the other is the Kistiakowsky's correlation using NBP (1).

For a monoacid TAG having 48 carbons such as tripalmitin (PPP) a single value of 39 Kcal/mol was reported, and 40 Kcal/mol for tristearin (SSS) having 54 carbons (6,7). These values were estimated by utilizing the Claussius-Clapeyron equation. Using the vapor pressure data reported by Perry et al (8) for a mixed TAG with 54 carbons, 2-oleyl-1,3-distearin (SOS), one may confirm the same value of  $\Delta H$ , 40 Kcal/mol. For the sake of simplicity and with the absence of relevant data,  $39.5 \pm 0.5$  Kcal/mol was selected as the representative value of  $\Delta H$  for TAG.

The NBP of triglycerides may be estimated from the plot of vapor pressure against temperature, in the form of  $\ln P$  vs  $1/T$ . The NBP of SSS and SOS were estimated from the vapor pressure plot to be about  $410 \pm 10$  ° C.

The next step is to estimate the NBP of sterols and gossypol, for which one may use capillary chromatograms of oilseed lipids along with TAG. Among many typical oilseed sterols (9),  $\beta$ -sitosterol, stigmasterol, (24-methylene)-cycloartanol, cycloartenol, and campesterol were considered in this report, all of which are the major sterols of  $\gamma$ -oryzanol found in rice bran oilseed (10). Typical GC capillary chromatograms of vegetable oil are made via either the direct on-line injection or the split injection. The direct injection method (11) provides data for compositional and boiling point information, mostly limited to TAG, whereas the split injection method with esterified oil extends the NBP information related to sterols and gossypol. A capillary chromatogram of cottonseed oil by the direct and the



split injection are given in Figures 1 and 2, and esters of rice bran oil by the split injection in Figure 3. In the direct injection of capillary GC, most of oilseed triglycerides elute at temperatures up to 360°C. Usually the TAG compounds completely elute within a short span of the elution temperature, a range of 10 to 15 ° C. As shown in Figure 1 the oilseed triglycerides were eluted in a few groups according to their carbon numbers in their acyl chains. The chromatograms in Figures 2 and 3 also showed that the sterols were also eluted at a narrow temperature span near diacylglycerols, and gossypol at a temperature near diacylglycerols. Using this information regarding the elution temperature, the NBP of gossypol and sterols were estimated about 40 ° C and 80 ° C lower than TAG, respectively. The estimation of the boiling point from gas chromatograms has been reported elsewhere (12).

The Kistiakowsky's equation, given below, may now be utilized.

$$\Delta H/T_b = K_1 + R \log_{10} T_b \quad (5)$$

where  $T_b$  denotes the normal boiling point in ° Kelvin, and  $K_1$  a constant.

With the  $T_b$  determined from the vapor pressure data of TAG, the value for the constant  $K_1$  was determined from equation (5) to be  $54 \pm 0.5$ . For the sake of simplicity, the specific gravity of gossypol and sterols were assumed to be equal to that of simple TAG such as SSS or OOO as reported in the literature (13).

Using the range of  $\Delta H$  values obtained by equation (5), the  $\delta$  values of sterols and gossypol were calculated by equation (2), as summarized in Table 2.

#### Prediction of Extraction Performance

As seen from Table 2, the estimated values of  $\delta$  for the alkane solvents having 5 to 7 carbons are practically invariant, whereas those  $\delta$  values of alcohols vary significantly. This indicates that the alkanes solvents have about the same solubility characteristics, while the alcohol solvents may show behaviors quite distinctive from the hydrocarbon solvents. The difference in the pattern of  $\delta$  between the alcohols and the paraffinic solvents is expected, which may be mainly attributed to the difference in the physical properties. The estimated values of solubility parameter indicate that the alkanes are excellent solvents for TAG, but not for gossypol or sterols. This may be clearly understood, when one compares the differential values of  $\delta$  between TAG and the alkanes with those between TAG and the alcohols.

Now that the values of solubility parameter for gossypol and sterols are available, one may use this information to select or screen the alternative solvent system for better extraction of these components.

Gossypol is known to be difficult to extract using alkane solvents, but easily extracted by alcohols (14). To extract gossypol and TAG at the same time, cottonseed extraction has been

tested by using either ethanol (15) or iso-propyl alcohol (16). As can be projected from the solubility relationship given in Table 2, the alcoholic solvents were proved to be a poor solvent to extract the main species of oilseed lipid, TAG. It was concluded that because greater amounts of alcohol than hexane were required to achieve the extraction goal, the extraction process using 100% EtOH or IPA was economically inviable. The same conclusion was clearly realized from the analysis of solubility parameters given in Table 2.

To balance the shortcomings of the types of solvents, mixtures of alcohol with alkanes were tried for extracting gossypol and TAG (17). As shown in Table 3, what was really required for gossypol extraction was not 100% alcohol solvent, but a small fraction of alcohol in the mixture solvent of alcohol-alkane.

The extraction yield using 100% iso- or n-hexane was 26-27%, whereas extraction using alcohol-hexane mixtures yielded 28-29%. The increase in the extraction yields remained in the limit of experimental variation. The extraction yield information indicated that the mixed solvent system was as efficient as the hydrocarbons in the recovery of TAG. The main advantage of the mixed solvent was that the toxic component, especially free gossypol, was removed by extraction with a very high efficiency by simply having 10-25% added to alkanes. The estimated values of the solubility parameter fall between 6.5 to 8.5. The details of extraction with the mixed solvent was presented elsewhere (17).

Since the values of the solubility parameter of SC-CO<sub>2</sub> are variant (See equation (2)), extraction of some species of oilseed lipids such as sterols may possibly be affected by the density of extraction medium in supercritical state. According to a recent report on the rice bran extraction using SC-CO<sub>2</sub> (18), the composition of oil, especially the sterol content, was affected by the values of Hildebrand's solubility parameter for the extraction medium. As shown in the capillary chromatograms of rice bran oil (Figures 3 and 4), the amount of sterol extracted using SC-CO<sub>2</sub> at 7000 psi was less than that of SC-CO<sub>2</sub> at 9000 psi and 100 °C. The range of the solubility parameters for SC-CO<sub>2</sub> at the two conditions was estimated to be 8.5-9 by equation (2). The integrated area ratio of sterol peaks to the internal standard (IS) in Figure 4 was about twice as large as those found in Figure 3. These results indicated that the selective extraction of specific group compounds of oilseed lipids should be possible by regulating the  $\delta$  of SC-CO<sub>2</sub>. This observation is consistent with the findings of Czubryt et al (19) that the solubility of fatty acids in carbon dioxide, particularly a fatty acid, is dependent upon the state of SC-CO<sub>2</sub>.

### Conclusion

In summary the following were reported in this communication.

(1) A method of estimating Hildebrand's solubility parameter was demonstrated for typical oilseed solvents and lipids. (2) The



estimated values of the solubility parameters were used to predict and demonstrate that selective extraction of specific lipids should be possible either via multi-component solvents or SC-CO<sub>2</sub>.

The precision of the estimated values may be improved with the experimental determination of physical properties.

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TABLE 1. Physical properties of alternative Solvents

compound	mw	NBP (°C)	P <sub>c</sub> (Atm)	$\rho^1$	$\Delta H^1$	$\delta^1$
n-C <sub>5</sub>	72.2	36	33	0.62	6.2	6.8
n-C <sub>6</sub>	86.2	68	30	0.65	6.9	6.5
i-C <sub>6</sub>	86.2	60	30	0.65	6.6	6.4
n-C <sub>7</sub>	100.2	98	27	0.68	7.6	6.6
MeOH	32	65	78	0.79	8.4	14
EtOH	46	78	63	0.79	9.3	11
IPA	60	80	53	0.78	9.5	10.5

1.  $\rho$  in (g/cc),  $\Delta H$  in (Kcal/Mole) and  $\delta$  (cal/cc)<sup>1/2</sup>.



Table 2. Comparison of the estimated values of Hildebrand's solubility parameter( $\delta$ ) for alternative solvents and oilseed lipids.

Compound	$\delta$ (cal/cc) <sup>1/2</sup>
n/i - Hexane	6.4/6.5
n - Heptane	6.6
TAG	6.5~7.5
EtOH	11
IPA	10.5
Gossypol	8.5 ~ 9
Sterols	8.5 ~ 9
SC-CO <sub>2</sub> <sup>1</sup>	6 ~ 11

1. Estimated using equation (2).

Table 3. Oil and Gossypol Extraction Performance with  
Hydrocarbon Solvent Mixtures with Alcohol

Base Hydrocarbon(%)		Alcohol Type, Vol%	Extraction Oil Yield <sup>1</sup> (%)	Gossypol (%)	
				Total <sup>2</sup>	Free <sup>2</sup>
i-Hexane,	100	none	26.2	1.30	0.95
i-Hexane,	90	EtOH, 10	26.7	0.41	0.18
i-Hexane,	75	EtOH, 25	28.0	0.60	0.11
i-Hexane,	90	IPA, 10	27.5	0.50	0.26
i-Hexane,	75	IPA, 25	29.0	0.45	0.12
n-Hexane,	100	none	27.0	1.10	1.05

1. An average value with a SD of  $\pm 5\%$ .

2. Raw cottonseed flakes contained 1.05 % total and 1.03 % free gossypol, determined by the AOCS official methods.

## Captions for Figures

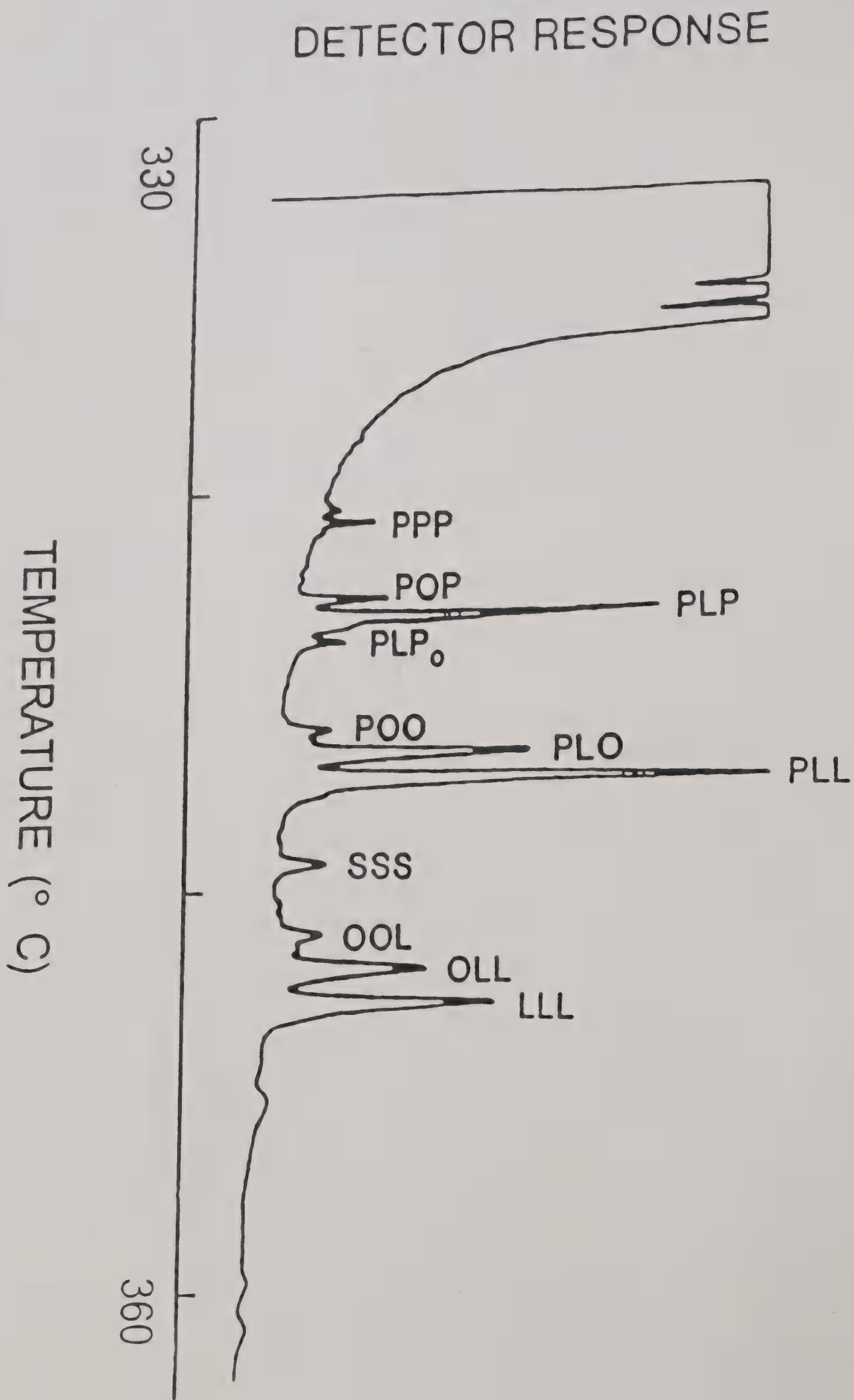
Figure 1. A capillary chromatogram of cottonseed oil triglycerides with the direct cold-on column injection, temperature programmed between 340-360 ° C. Hydrogen was used as carrier gas at 1 ml/min. A microbore TAP column, 25 m x 0.25 mm (Chrompac, Raritan, NJ). More details on GC conditions given elsewhere (14).

Figure 2. A capillary GC analysis of gossypol and oilseed TAG. (Chromatographic details identical to Fig.1)

Figure 3. Gas chromatogram of SC-CO<sub>2</sub> extracted rice bran oil (T.S.-derivatized) with SC-CO<sub>2</sub> at 7000 Psi and 80 ° C. More details given in reference (20).

Figure 4. Gas chromatogram of SC-CO<sub>2</sub> extracted rice bran oil (T.S.-derivatized) with SC-CO<sub>2</sub> at 9000 Psi and 100 ° C. GC conditions identical to Fig 3.

Figure 1





*PROCEEDINGS*

# 46th Oilseed Conference

**Processing Efficiency:  
Meeting the Challenge**

**March 9–11, 1997**

**Hotel Monteleone**

**New Orleans, Louisiana, USA**

Co-Sponsored by:

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The National Cottonseed  
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# **46th Oilseed Conference**

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## **Executive Committee:**

General Chairperson: **Robert C. Edmondson**  
Applied Engineering & Science

Technical Chairperson: **Khee C. Rhee**  
Food Protein R & D Center, Texas A&M University

Poster Session Chairperson: **John Patrick Jordan**  
Southern Regional Research Center/ARS/USDA

Tabletop Exhibit Chairperson: **Richard T. Gadomski**  
PSI Group of Companies

## **Organizing Committee:**

**Donald E. Britton**, Mid-Continent Labs  
**Edward Campbell**, Archer Daniels Midland Co.

**Roy A. Carr**, POS Pilot Plant Corp.  
**William G. Clark**, Yazoo Valley Mill, Inc.  
**Edith Conkerton**

**Steve Cooper**, Osceola Products  
**Walter R. Farr**, Owensboro Grain Co.

**Lance A. Forster, Jr.**, National Cottonseed Products Association, Inc.  
**Daniel P. French**, French Oil Machinery Co.

**Timothy D. Gum**, Applied Engineering & Science  
**Lynn A. Jones**, National Cottonseed Products Association, Inc.  
**Juan Kindelan**, PSI Process Systems Inc.

**Mohammad Jaafer Ahmad**, PORIM  
**Larry Johnson**, Center for Crops Utilization Research, Iowa State University  
**Michael T. Murray**, Alfa Laval Separation Inc.

**Deland J. Myers**, Department Food Science & Human Nutrition, Iowa State University  
**Zivko Nikolov**, Center for Crops Utilization Research, Iowa State University  
**Armand Pepperman**, Southern Regional Research Center/ARS/USDA  
**Shirley Saucier**, Southern Regional Research Center/ARS/USDA  
**Robert L. Stroup**, French Oil Machinery Co.  
**Peter J. Wan**, Southern Regional Research Center/ARS/USDA

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*DEDICATION*

# 46th Oilseed Conference

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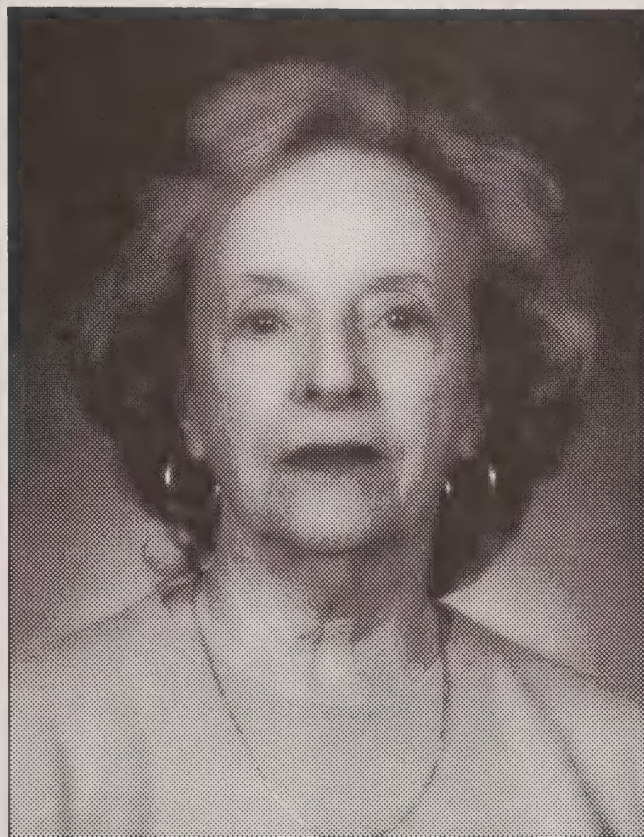




# 46th Oilseed Conference

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## DEDICATION

The 46th Oilseed Conference is dedicated to Shirley T. Saucier. Her name first appeared as the conference coordinator for the old "Cottonseed Processing Clinic" in 1967. She has been deeply involved with every annual session of this meeting up through the present event. Such an outstanding record, spanning two employers (the USDA until 1985, and Jones Management Services since that time) and three different names (in 1971 it became the "Oilseed Processing Clinic" and in 1995 it received its present title) deserves recognition itself. However, in Ms. Saucier's case, her contribution is much more than longevity. Over the decades, she has acted as the capable organizer pulling all the threads together of an event run by committee and in the process has made every meeting a success and made every general chairman and every USDA staff co-chair look competent and organized. There are many who owe her a vote of thanks. Truly, the Oilseed Conference would not be the event it is today if it had not benefited from three decades of capable guidance by Shirley Saucier.

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# *PROGRAM SCHEDULE*

## 46th Oilseed Conference

**Processing Efficiency:  
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**Hotel Monteleone**

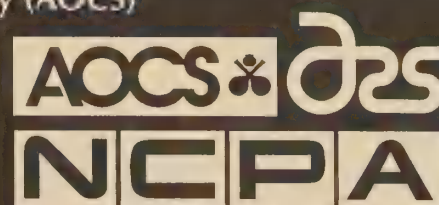
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# ***46th Oilseed Conference***

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## PROGRAM SCHEDULE

### Sunday, March 9, 1997

12:00pm - 7:00pm

1:00pm - 5:00pm

6:00pm - 7:00pm

Registration

Table Top Exhibit Set Up

Opening Reception

Foyer: Queen Anne Room

La Nouvelle Orleans East/West

La Nouvelle Orleans East/West

### Monday, March 10, 1997

7:30am - 5:00pm

8:30am - 9:15am

Registration

Opening Remarks:

Foyer: Queen Anne Room

Queen Anne Room

#### INVOCATION

**David H. Kinard**

National Cottonseed Products Association

Memphis, TN

#### CALL TO ORDER BY GENERAL CHAIRPERSON

**Robert C. Edmondson**, Senior Vice President

Applied Engineering & Science

Atlanta, GA

#### DEDICATION OF MEETING

**John Patrick Jordan**, Director

USDA, ARS, Southern Regional Research Center

New Orleans, LA

#### KEYNOTE PRESENTATION:

*The Evolution of the Oilseed Industry and Outlook for the Future*

**John C. Baize**, President

John C. Baize and Associates

Falls Church, VA

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## SESSION I

Monday, March 10, 1997

9:15am - 12:00Noon

Queen Anne Room

Session Chairperson: **William G. Clark**

Yazoo Valley Oil Mill, Inc.

Greenwood, MS

*Benchmarking to Achieve World-Class Performance*

**Bill Windle**

A.T. Kearney

Chicago, IL

*Minimizing Oil Loss in Miscella Refining*

**Stan C. Loft**

Loft Consulting Services, Inc.

San Diego, CA

*Minimizing Oil Loss in Miscella Refining*

**Timothy G. Kemper**

French Oil Mill Machinery Co.

Piqua, OH

*Poster Introductions*

**Armand Pepperman**

USDA, ARS, Southern Regional Research Center

New Orleans, LA



## LUNCHEON

Monday, March 10, 1997

12:00pm - 1:45pm

La Nouvelle Orleans East/West

*dedicated time to view poster presentations and visit with table top exhibitors*

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## SESSION II

Monday, March 10, 1997

2:00pm - 5:00pm

Queen Anne Room

Session Chairperson: **Lynn A. Jones**  
National Cottonseed Products Association  
Memphis, TN

*National Emission Standards for Hazardous Air Pollutants for Vegetable Oil Production Facilities*

**James F. Durham**  
Environmental Protection Agency  
Research Triangle Park, NC

*Proposed Changes to the NAAQS for PM and Ozone*

**Pat Delamater**  
Trinity Consultants Incorporated  
Dallas, TX

*New Technology of Plant Automation*

**Ronnie Sieber**  
Lubbock Electric  
Lubbock, TX

*Today's Research...Tomorrow's Impact*

**John Patrick Jordan**  
USDA, ARS, Southern Regional Research Center  
New Orleans, LA

*Update on Methods to Prevent Aflatoxin Formation*

**Peter J. Cotty**  
USDA, ARS, Southern Regional Research Center  
New Orleans, LA

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## SESSION III

Tuesday, March 11, 1997

9:00am - 12:00Noon

Queen Anne Room

Session Chairperson: **Walter E. Farr**

Owensboro Grain Company

Owensboro, KY

*New Technology Development at Texas A&M University*

**Steve R. Gregory**

Texas A&M University, Food Protein R&D Center

College Station, TX

*Whole Cottonseed Research & Promotion Program at Cotton Incorporated*

**T.C. Wedegaertner**

**W.F. Lalor**

Cotton Incorporated

Raleigh, NC

*New Technology Development - Genetics*

**Phil Kerr**

DuPont Quality Grains

Des Moines, IA

*Biotechnology in the Oilseed Industry*

**Kathleen Warner**

USDA, ARS, National Center for Agricultural Utilization Research

Peoria, IL

*Food Oil Substitutes - Frying Applications*

**William E. Artz**

The University of Illinois

Urbana, IL



## LUNCHEON

Tuesday, March 11, 1997 .

12:00pm - 1:45pm

La Nouvelle Orleans East/West

Featured Presentation: *Washington Outlook*

presented by: **David A. Bossman**, President

American Feed Industries Association

Arlington, VA

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## **SAFETY MANAGEMENT WORKSHOP: ITS IMPORTANCE AND HOW TO GET STARTED**

Tuesday, March 11, 1997

1:30pm - 3:30pm

Queen Anne Room

Session Moderator: **C. Louis Kingsbaker, Jr.**  
C.L. Kingsbaker, Inc.  
Atlanta, GA

*Rethinking Your Safety Program*

**Raymond Rush**

Safety and Industry Consultant

Jackson, MS

**John E. Robson**

Bunge Corporation

Destrehan, LA

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*POSTER SESSION*

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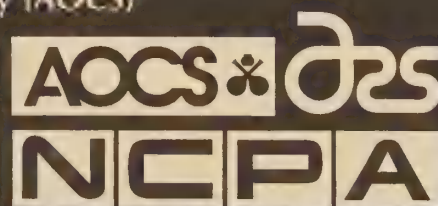
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## POSTER SESSION

Sunday, March 9, 1997 - 6:00pm-7:00pm

Monday, March 10, 1997 - 12:00Noon-1:45pm

La Nouvelle Orleans East/West

Session Chairperson: **Armand Pepperman**  
USDA, ARS, Southern Regional Research Center  
New Orleans, LA

(presenting authors listed in **bold**)

*Supercritical Fluid Extraction of Oil from Oilseed - An AOCS/AOAC Collaborative Study*

David L. Berner, Leslie J.D. Myer and **J.D. Colburn**

Isco Inc.

Lincoln, NE

*Characterization of Soapstocks from Corn Germ and Peanut Oil Refining*

**Michael K. Dowd**

USDA, ARS, Southern Regional Research Center

New Orleans, LA

*Characterization of Eleostearic Acid Production in Tung Nut Homogenates*

**John M. Dyer**, Fuqiang Tang, Dorselyn Chapital, Alan R. Lax, Hurley S. Shepherd, Sing Shih  
and Armand Pepperman

USDA, ARS, Southern Regional Research Center

New Orleans, LA

*Improving Oil Yields and Co-Products Values by Sequential Extraction Processing of Corn*

M.P. Hojilla-Evangelista, **L.A. Johnson**, and D.J. Myers

Center for Crops Utilization Research and Dept. of Food Science and Human Nutrition

Iowa State University

Ames, IA

*Estimation of Hildebrand's Solubility Parameter for Oilseed Lipids and Solvents*

**M.S. Kuk**

USDA, ARS, Southern Regional Research Center

New Orleans, LA

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*A Recombinant Enzyme from Transgenic Soybeans: Processing and Extraction*

**Ann Kusnadi, Roque Evengelista and Zivko Nikolov**

Department of Agricultural and Biosystems Engineering, Department of Food Science and  
Human Nutrition, Center for Crops Utilization Research

Iowa State University

Ames, IA

*Two New Methods for Producing Epoxidized Oils for Industrial Uses*

**George J. Piazza, Alberto Nuñez, Philip E. Sonnet, and Thomas A. Foglia**

Eastern Regional Research Center, ARS, USDA

Wyndmoor, PA

*Soapstock Composition and Its Impact on the Quality of Cottonseed Meal*

**P.J. Wan, M.K. Dowd, and D.E. Britton**

USDA, ARS, Southern Regional Research Center, New Orleans, LA

Mid-Continent Laboratories, Inc., Memphis, TN

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# *TABLE TOP EXHIBITS*

## 46th Oilseed Conference

Processing Efficiency:  
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Processing Efficiency:  
Meeting the Challenge

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# 46th Oilseed Conference

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## Table Top Exhibits

**Alfa Laval Separation Inc.**, 200 South Park Boulevard, Greenwood, IN 46143. The fats and oils business unit of Alfa Laval Separation focuses its efforts on the sales of new centrifuges and mixers for vegetable oil processing, customer service (field service, parts, repairs), and separator-based process engineering.

**Alltech Biotechnology, Inc.**, 3031 Catnip Hill Pike, Nicholasville, KY 40356. Alltech Biotechnology, Inc. produces antioxidants for stabilizing oils and fats of various origins. Product lines include natural and traditional antioxidants. Alltech also supplies many other additives to feed and pet food industries, rendering facilities and brewing and distilling companies in over 60 countries.

**Applied Engineering & Science**, 2261 Perimeter Park Drive, Atlanta, GA 30062. Engineering and construction management in oilseeds, fats and oil and corn processing; environmental, processing facilities.

**Bird Machine Company**, 100 Neponset Street, S. Walpole, MA 02071-9103. Bird Machine Company manufactures liquid/solid separation equipment. Bird is ISO 9001 certified and is able to offer solutions for processing to the oil seed industry. Stop by our booth to learn more.

**Continental Air Systems**, P. O. Box 400, Winfield, AL 35594. The Outr-A-Vac Drum filter offers maximum filtering efficiency at lower equipment cost, lower horsepower, less space, lower pressure drop, and less maintenance cost than other dry air filtering devices. Product laden air is cleaned as it passes through filter media on a rotating drum and clean air exits from the drum interior.

**Crown Iron Works Company**, P. O. Box 1364, Minneapolis, MN 55440. Crown Iron Works specializes in design and engineering for oilseed and oil processing. Crown supplies traditional and specialty extraction equipment along with refining systems for edible oils and derivatives including methyl esters, fatty acids and glycerine. Crown has offices located in the U.S.A., United Kingdom and Central America.

**De Smet Process & Technology**, 2839 Paces Ferry Road, Suite 880, Atlanta, GA 30339. The De Smet Group will present its full range of oil-related equipment and engineering services, including oilseeds milling and extraction, oils and fats refining and modification, process engineering and safety and environmental engineering.

**Ferrell-Ross**, 805 S. Decker Drive, Bluffton, IN 46714. Ferrell-Ross offers a complete line of cleaning, cracking, and flaking equipment. New developments will be featured in process control (weigh systems), precision cleaning (Clipper oilseed cleaner), fine grinding (flour mill), and roll resurfacing (Accu-Tram). Ferrell-Ross also offers service and equipment maintenance.

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**French Oil Mill Machinery Company**, 1035 W. Green Street, Piqua, OH 45356. The French Oil Mill Machinery Company offers a complete line of modern and efficient oilseed preparation and solvent extraction equipment and process engineering services. The new Reflex™ extractor.

**Hi Roller Conveyors**, 5100 West 12th Street, Sioux Falls, SD 57107-0514. Hi Roller conveyors are ■ totally enclosed, self-cleaning, and self-reloading belt conveyor. All bearings are external. All dust or spilled material is contained, reloaded, and discharged with the product. Conveyors are designed for 24 hour service and for capacities to 100,000 bushels per hour.

**Industrial Metal Products Division of Continental Eagle Corporation**, P. O. Box 21212, Phoenix, AZ 85036. A manufacturer of oilseed preparation machinery for various oilseeds, specializing in the preparation of cottonseed. Serving the oilseed industry for 44 years. IMPCO systems are known worldwide for quality manufacture and ease of process operations. Product line includes the LE-176 Saw Linter, Lint Cleaners, Decorticator/Separator & Hull Beating Systems.

**Intersystems**, 13330 I Street, Omaha, NE 68137. Intersystems will feature En-Masse Conveyors, Bucket Elevators, Automatic Truck Probes, Automatic Samplers and Kleen-Masse Conveyors. Kleen-Masse Conveyors represent a patented new design for product cleanout. Equipment can be manufactured from galvanized stainless steel, or hot roll steel.

**Lubbock Electric Company, Inc.**, 1108 34th Street, Lubbock, TX 79405-1799. Lubbock Electric Company, provider of complete PLC and Mmi systems design and integration, Automation, Process Control, and SCADA Systems, U.S. Data FactoryLink, Wonderware, and Taylor Mmi Software for operator interface, Total Control Products Operator Interface, Panel and Console Fabrication, and Start-up services.

**N. Hunt Moore & Associates, Inc.**, 3951 Senator Street, Memphis, TN 38118. N. Hunt Moore & Associates is celebrating its 45th anniversary of serving the oilseed industry by offering quality equipment from Roskamp Champion, Tecnal, Krupp, Frannino and Escher Wyss. N. Hunt Moore performs engineering services including layout, process control and PSM (PHA audits).

**Oil-Dri Corporation of America**, 410 North Michigan Avenue, 4th Floor, Chicago, IL 60611. Fluids Purification Group, a division of Oil-Dri Corporation of America, will feature our family of specialty adsorbents. These products include Pure-Flo® Supreme bleaching adsorbents for the purification of fats, oils, and oleochemicals, and Select™ 350 selective adsorbent for the removal of soaps, metals and phospholipids.

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**Plant Maintenance Service Corporation**, 3000 Fite Road, P.O. Box 280883, Memphis, TN. Fabrication, installation and maintenance, specializing in oilseed processing equipment. Pressure vessels, heat exchangers, condenser, and evaporators per ASME code. Conveyors, hoppers and bins furnished and installed. Emergency services and plant turn arounds also provided.

**PSI Process Systems, Inc.**, 1790 Kirby Parkway, Suite 300, Memphis, TN 38138. PSI representatives will present their experience in design, engineering and construction of state-of-the-art vegetable oil processing and refining plants. Emphasis will be on safety in design and construction, control systems, procurement, and single source responsibility for design/build or turnkey projects.

**Roskamp Champion**, 2975 Airline Circle, Waterloo, IA 50703. Roskamp Champion is the world leader in oilseed preparation equipment. We manufacture flaking and cracking mills, dehullers and hammermills that are energy efficient, robust and reliable. Roskamp Champion offers service and support worldwide.

**Tramco, Inc.**, 1020 East 19th Street, Wichita, KS 67214. Tramco will exhibit literature on the "World's Most Complete Line of Chain Conveyors" for the processing industry.

**Trumbo, Inc.**, 1106 Kansas, Memphis, TN 38106. ASME pressure vessels, heat-exchangers, evaporators, storage tanks (shop and field erected), process piping, miscellaneous structural steel towers, catwalks and supports, mechanical contracting, boiler repairs and alterations, sterilizers with patented automatic doors, licensed for Phillips Rod Baffle Heat-Exchanger

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*The Evolution of the Oilseed Industry and  
Outlook for the Future*

**John C. Baize**

John C. Baize and Associates

Falls Church, VA

# 46th Oilseed Conference

**Processing Efficiency:  
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# **The Evolution of the Oilseed Industry and Outlook for the Future**

**By John C. Baize  
President  
John C. Baize and Associates**

**46<sup>TH</sup> Oilseed Conference  
New Orleans, Louisiana  
March 10, 1997**

Good morning ladies and gentlemen. It is a very distinct pleasure for me to be able to attend the 46<sup>th</sup> Oilseed Conference and an honor to be asked to address the subject of the evolution of the global oilseed industry and the outlook for the industry's future. I regret I will be unable to stay for the entire conference because of previous commitments, but wish you a very successful meeting.

I think it is important to consider for a moment how far the oilseeds industry has come in a very short period of time. While soybeans have been consumed in Asia for thousands of years and cotton has been the fabric of our lives for almost as long, it is only in this century that vegetable oils and protein meals have been an essential element of the very existence of mankind. Consider that 50 years ago most foods were cooked using animal fats. Sophisticated animal feeding using balanced rations was unheard of. Poultry meat made up a very small part of the diet.

All of that changed in the middle of this century with the beginning of large-scale production of soybeans and the development of solvent extraction to separate soybeans, cottonseed, sunflowers, and other oilseeds into vegetable oils and protein-rich meals. This made it possible for the widespread production of margarine and shortening to replace animal fats and for the development of the efficient swine and poultry sectors that are the dominant providers of dietary protein to the world today. I contend that in many ways the development of the modern oilseed production, processing, vegetable oil refining, and animal feed industries in this century has been as important as any other factor in shaping the world we live in today. Think about how different our society would be without these industries.

I recognize that this meeting largely is focused on the cottonseed industry. However, it is the soybean sector that dominates the U.S. and global oilseed industry. As goes the soybean sector so goes the cottonseed, sunflowerseed, and other oilseed sectors. Therefore, with apologies to those of you in the cottonseed processing sector, the bulk of my remarks today will be focused on the soybean sector.

The U.S. soybean industry was a very small industry 60 years ago. In 1935 the U.S. produced only 2.79 million hectares (6.9 million acres) of soybeans, but only 1.17 million hectares (2.9 million acres) were harvested. The remainder were used as hay or as a green manure. Total soybean production was only 1.3 million metric tons (mt) or 48 million



bushels. By the end of World War II plantings had almost doubled to 5.26 million hectares (13 million acres), but harvested area had more than tripled to 4.25 million hectares (10.5 million acres). Production reached 5.25 million mt (193 million bushels) in 1945. More soybeans are now produced in each of four states -- Illinois, Iowa, Minnesota, and Indiana -- than were grown in the entire country in 1945.

The soybean industry continued its fantastic growth in area after World War II until it reached its high of 28.7 million hectares (71 million acres) planted in 1979. However, after 1979 the soybean industry began a long, sustained decline in planted area. This occurred because of several factors including low prices, increased disease pressure, counter-productive farm policies, and increased competition for land from other crops. The Farm Bill of 1985 hit the soybean industry hard because it established the Conservation Reserve Program as well as severely limited farmers' ability to shift land out of corn, cotton, and other program crops into soybeans. By 1990 U.S. soybean planted area fell to a modern low of only 23.4 million hectares (57.8 million acres), 18 percent lower than 11 years earlier. Fortunately, because of greater profitability and less restrictive farm policies, the soybean sector recently has begun to regain some of the area it lost in the 1980s. U.S. farmers planted 26 million hectares (64.2 million acres) of soybeans in 1996, the highest since 1979. Some analysts believe plantings in 1997 may exceed 26.3 million hectares (65 million acres) because of the very positive ratio that currently exists between the price of soybeans and other crops.

Fortunately, the sharp decline in soybean plantings in the 1980s largely was offset by increased national average yields. The higher national yield came about because of the development of higher yielding varieties, a shift toward no-till farming, and plantings in narrower row widths. Another major reason for the higher yield was a decline in soybean plantings in the South and Southeast where average yields were low and a sustained increase in plantings in the higher-yielding Western Corn Belt. Between 1979 and 1994 soybean plantings in the South and Southeast fell from 9.98 million hectares (24.4 million acres) to only 4.57 million hectares (11.3 million acres), a drop of 54 percent. Plantings in the six states of the Southeast (Alabama, Georgia, Florida, South Carolina, North Carolina, and Virginia) fell by more than 62 percent. During this same period, plantings in the states of North Dakota, South Dakota, Nebraska, and Minnesota increased by 28.7 percent or more than 1.8 million hectares (4.5 million acres). Today more soybeans are planted west of the Mississippi River than east of it and the area planted to soybeans in the Western Corn Belt continues to expand.

The decline in soybean plantings in the Southeast has begun to pose problems for that region's huge poultry and swine sectors. Because of the high freight costs required to transport ever-increasing amounts of soybeans, soybean meal, and corn in from the Midwest, the Southeastern poultry sector has seen its cost advantages decline. It is for this reason that much of the new expansion in the poultry sector is in Tennessee, Kentucky, and other states closer to the major soybean and corn growing areas. Unless Southeastern soybean plantings and production begin to recover soon, I foresee this trend continuing. We also could see a situation where South American soybeans and soybean



meal begin to be imported during the spring and summer months into Savannah, Mobile, Charleston and other Southeastern ports to supply the Southeast's poultry and swine sectors' growing need for meal.

While U.S. soybean area and production were declining in the 1980s, foreign producers were sharply expanding their plantings. Argentina and Brazil expanded their combined soybean area and production from 10.8 million hectares and 18.75 mmt in 1979 to an estimated 18.2 million hectares and 35.6 mmt in 1996. This year the two countries are likely to produce a soybean crop of 40.7 mmt, an all-time high. Soybean area and production also have expanded in Paraguay, Bolivia, China, and other countries. In 1979 the U.S. soybean crop accounted for almost two-thirds of global soybean production. USDA estimates this year that the U.S. soybean crop will account for only about 49 percent of global soybean production. The U.S. is still the largest soybean producer by far, but it certainly does not enjoy the dominant position it once commanded.

If one looks at the growing global demand for protein meal and vegetable oils, it is clear that the rest of the world needed to expand oilseed production. During the last 30 years global vegetable oil and protein meal consumption have skyrocketed. This year USDA estimates global vegetable and marine oil consumption will total 70.1 mmt, an increase of 549% over the 10.8 mmt consumed in 1966. Global protein meal consumption has grown from 29.4 mmt in 1966 to over 143.8 mmt this year, an increase of 388 percent. This has come about not only because people all around the world have shifted from lard and tallow to vegetable oils and from butter to margarine, but also because of the urbanization of the world's population. Urban households often have two breadwinners and are more inclined to eat out in restaurants, particularly fast-food restaurants like McDonalds and Kentucky Fried Chicken (KFC). You are almost as likely to find one of these fast-food restaurants in Jakarta as you are in Milwaukee. Because fried foods are staples in such restaurants, demand for vegetable oils has skyrocketed. Yet, the world is far from saturated in vegetable oil. China and India, with a combined population of almost 2.4 billion people have a per capita vegetable oil consumption level of only about 10 kg. and 8.5 kg, respectively. That compares with 45.3 kg. in the United States. If India were raise its annual per capita consumption of vegetable oils to the 17kg. consumed in neighboring Pakistan, the additional oil required each year would be about 6.7 mmt. That's just slightly less than the 6.9 mmt of soybean oil consumed annually in the entire United States. Thus, it is very apparent that global demand for vegetable oils is far less than it will be in the future. Based on past trends, it is likely that the world will need at least an additional 20 mmt of vegetable oil per year in 2005 to meet consumer demand.

In recent years the driving force behind strong global demand for soybeans has been strong demand for soybean meal. Because of strong economic growth in Asia and Latin America, global demand for meat, poultry, and eggs is soaring. This, in turn, is leading to growing demand for soybean meal and other protein meals to feed the chickens, turkeys, swine, and other animals. Proof of the phenomenal growth in soybean meal demand is the fact that in this decade annual global consumption of soybean meal has grown by 20.8 mmt. That is equivalent to the total soybean meal content of the record crop of soybeans

Brazil is expected to produce this year. It truly is amazing that in 6 years global soybean meal demand has increased by an amount equaling the total output of the world's second-largest soybean producing country.

I suggest that the major event that is behind the phenomenal growth in global protein meal and vegetable oil demand was the collapse of the Soviet Union at the beginning of the decade. Prior to 1990 there were two alternative economic models in the world – capitalism and communism. With the economic collapse of the Soviet Union, communism effectively was destroyed as a workable economic model. Developing countries in Asia and elsewhere suddenly recognized that capitalism was the only economic system that was proven to work and they moved rapidly to liberalize their economies. China, India, Indonesia, the Philippines, and several other countries have acted rapidly to foster private sector economic growth, encourage foreign investment, and reduce external barriers to trade. Many countries have reduced import barriers faster than required by the Uruguay Round GATT Agreement. This has led to economic growth rates in several Asian countries of almost 10 percent per year and substantially higher in China. The global oilseeds sector is one of the first and greatest beneficiaries of this growth.

Consider what is going on in China. At the beginning of this decade China's government unleashed the forces of capitalism. During the 1990/91 marketing year China consumed about 1,300,000 mt of soybean meal and 758,000 mt of soybean oil. During the same year it exported 949,000 mt of soybeans and 1,268,000 mt of soybean meal. Since then things have changed radically. During the 1995/96 marketing year, China's total soybean meal consumption reached about 6,500,000 mt and its consumption of soybean oil was about 2,558,000 mt. Because of the huge increase in domestic demand for soybean meal and soybean oil, China has been forced to almost totally end its exports of whole soybeans and soybean meal, but also to begin major imports of these commodities. During the 1995/96 marketing year China imported approximately 1,500,000 mt of soybeans, 2,700,000 mt of soybean meal and 1,580,000 mt of soybean oil. Very quickly China has shifted from being a competitor to U.S. soybean farmers and processors into being one of their best customers.

I see nothing on the horizon, short of political or economic chaos within China, that will keep China from very soon becoming the world's largest net importer of soybeans and soybean meal as well as vegetable oils. It probably would trigger a revolution in China if the government were to cut off imports of foods wanted by its citizens. For China to raise its per capita vegetable oil consumption to just half of the 40 kg. per capita consumed in Taiwan, the additional vegetable oil required each year would be about 15 million mt or about the same amount of palm oil consumed in the entire world during the 1995/96 marketing year. Likewise, if China increases its per capita consumption of soybean meal to just half that of neighboring Taiwan, the additional soybeans needed annually will be greater than current U.S. soybean production. China is certain to be one of the dominant factors affecting global oilseed, protein meal, and vegetable oil markets in the future.



If one assumes that global demand will grow in the future as I have outlined, the question that begs to be addressed is whether or not the United States and the rest of the world will be able to produce the oilseeds and vegetable oil that will be needed. I am convinced that it will. In the case of vegetable oil, palm oil is certain to supply much of the future growth in vegetable oil demand. Global palm oil production has increased from only about 1,190,000 mt in 1966 to an estimated 16,169,000 mt in 1995/96. Most of the production expansion has been in Malaysia where palm oil output has increased during the period from 226,000 mt in 1966 to an estimated 8,260,000 mt during the 1995/96 marketing year. However, it is Indonesia that now is showing the greatest increase in palm oil output as new plantations come on stream in Sumatra, Kalimantan, and other islands. Scarcely a month goes by where some new major oil palm plantation expansion is not announced. This year Indonesia's palm oil production is expected to reach about 4,750,000 mt, but it is likely that Indonesian production will exceed that of Malaysia sometime before 2010. Should investors ever concentrate on growing oil palm in the Amazon Basin in Brazil, a region with similar latitude to Malaysia, there is virtually no limit to the palm oil that can be produced.

Rapeseed production also has an great potential to expand when Russia and Central Europe get their economies under control. Poland, Russia and other countries in the region have a great potential to expand rapeseed or canola production. The unknown is whether the oil produced from the rapeseed will be consumed in the region or exported elsewhere. Certainly most of the rapeseed meal will be consumed in the region to produce the meat the region needs and will consume under a more stable economic environment. Sunflowerseed output in Central Europe, Russia and other CIS (Commonwealth of Independent States) also can and will be expanded greatly in the future, in my opinion..

No doubt global cottonseed production will expand in the future as a result of increased area and higher yields. The Ataturk Dam in Turkey should allow that country to expand cotton production. I suggest Brazil also should contribute and greater share of the world's cotton lint and cottonseed production in the future. A major unknown is how much cotton production growth can be expected in the United States in the face of strong competition from other crops in a decoupled farm policy environment. A lot of farmers in the Delta that grew 150 bushel per acre corn last year are not inclined to expand their cottons plantings considering the far lower risk they have growing corn. However, if the boll weevil eradication program is successful in the Delta, cotton should stage a comeback there.

No doubt China has the capability to expand its cotton yields with new varieties and better insect and disease control. However, the amount of farmland in China is shrinking as a result of increased urbanization. There is intense competition from a wide array of crops for the land that is left. China's cotton area has declined by about 30 percent in the last 5 years and appears to still be declining. It will be hard for Chinese cotton yields to offset likely declines in cotton plantings.

I contend that there is a great potential to expand soybean production in the U.S, and

elsewhere as demand expands. The 1996 farm bill makes it possible for soybeans to equitably compete with other crops for land in the United States for the first time in a decade and strong protein and oil demand most likely will allow plantings to expand. Large tract of land in the Cerrados region of Brazil can and will be planted to soybeans if world prices and infrastructure development make it profitable. This year soybeans are being shipped for the first time from the Brazilian state of Mato Grosso by barge on the Tapajos River for loading on ships on the Amazon. This promises to cut transport costs substantially and improve farmer prices as well. The Inter-American Development Bank presently is conducting a feasibility study for the construction of a railroad from the Mato Grosso city of Cuiba across Bolivia to Chile's port of Arica on the Pacific. If such a railroad were to be built it would substantially reduce the time and cost of shipping Brazilian soybeans to the growing Asian market.

I suggest also that we are now just now beginning to see tip of the iceberg with respect to breakthroughs in increased soybean yields stemming from bio-genetics. Roundup-Ready™ soybeans are the first of scores of bio-engineered soybean varieties likely to reach the market with increased yields and production lower costs. The next generation of soybean varieties will offer enhanced intrinsic traits such as higher monosaturates, higher protein, enhanced amino acid profiles, and other desirable traits. Because soybeans are so widely grown and thus far unable to be hybridized, they are the crop that may offer the greatest financial incentive to bio-engineering firms developing new crop varieties. While Greenpeace and other groups have raised a stink in Europe about Roundup-Ready™ soybeans, there is no evidence that there is any real market resistance to their use. We may face greater problems when new bio-engineered varieties containing different compositions reach the market, but I suggest in the long run Europe and the rest of the world will have no practical alternative but to accept and use them.

Soybean yields also should receive a substantial boost in the future from bio-engineering and traditional breeding programs. The U.S. had a record soybean yield of 2.781 mt/hectare (41.4 bushels/acre) in 1994. This was far higher than the previous level of 2.53 mt/hectare in 1992. Even the flood- and drought-impacted 1996 U.S. soybean crop had a yield of 2.527 mt/hectare. I see no reason the U.S. cannot achieve an average soybean yield of 3.0 mt/hectare (45 bushels per acre) in the near future and it may not be long before a 3.3 average yield is reached. A 26.7 million hectare (66 million acre) planted area with a 3 mt/hectare yield would produce a crop of 80 million mt (2.95 billion bushels). In fact, it is the goal of the U.S. soybean industry to produce a 3 billion bushel crop (81.6 million mt) by 2005. This amount certainly will be needed by the world since trend-line projections show the world will need an additional 35 million mt (1.29 billion bushels) of soybeans by 2005 to meet growing meal and oil demand.

One the best things the soybean industry has going for it is the National Soybean Checkoff. The checkoff annually is raising many millions of dollars which are being invested in production and utilization research, new uses development, and market development here and abroad. This major investment by the nation's soybean farmers is



beginning to pay big dividends and is certain to continue to do so in the future. Farmers' checkoff investments are proving to be a very sound investment.

If one accepts that what I have reviewed today is on the market, it is hard to believe the future for the global oilseed industry is not bright. No doubt there will be periods when prices and processing margins are not so good, but overall I foresee a bright, profitable future.

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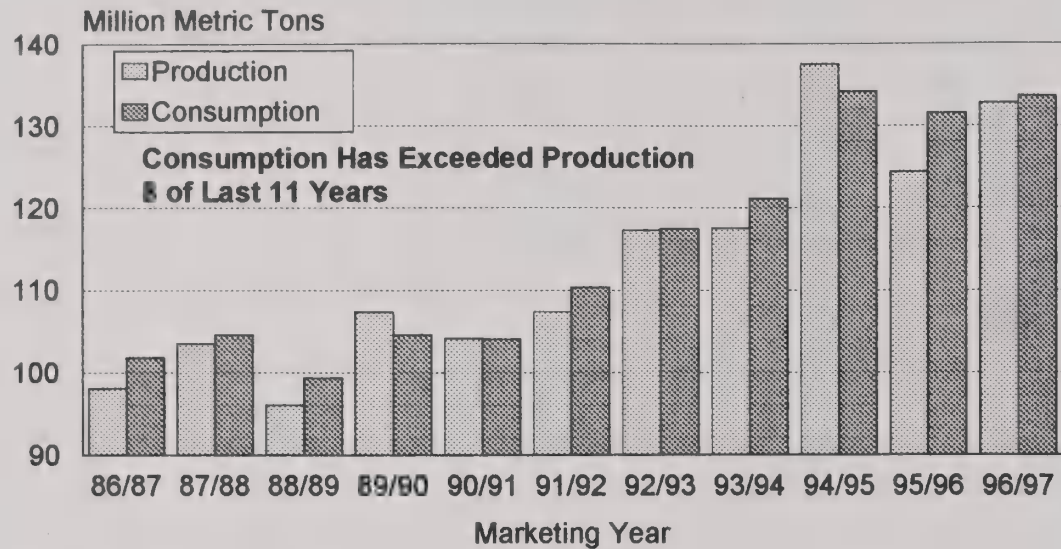
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Fax: 703-698-0321

E-Mail: [JBAIZE@msn.com](mailto:JBAIZE@msn.com)

## Global Production and Consumption of Soybeans

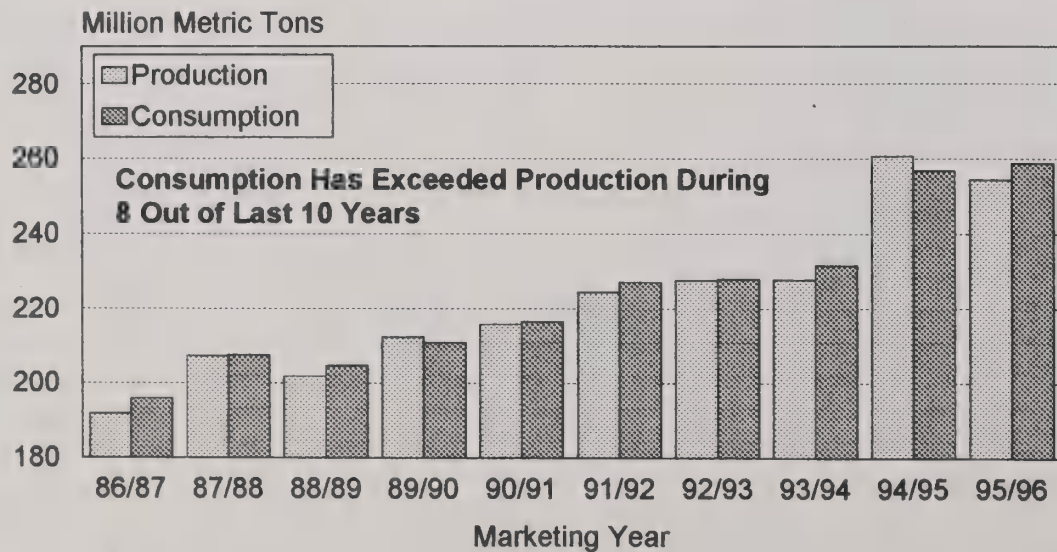
1986/87 - 1995/96



Source: USDA, February 1997

## Global Production and Consumption of All Oilseeds

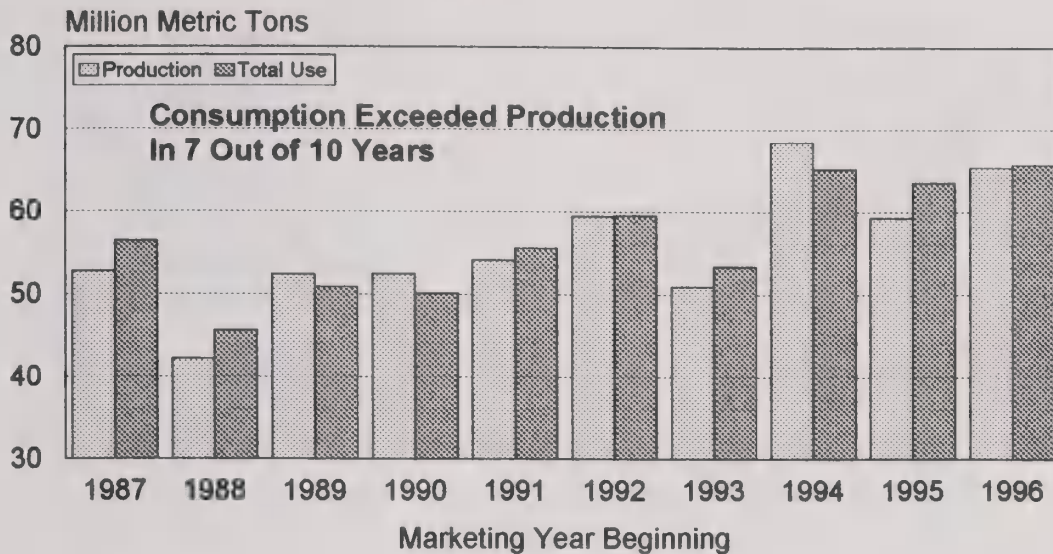
1986/87 - 1995/96



Source: USDA/NASS, November 1996

## U.S. Production and Consumption of Soybeans

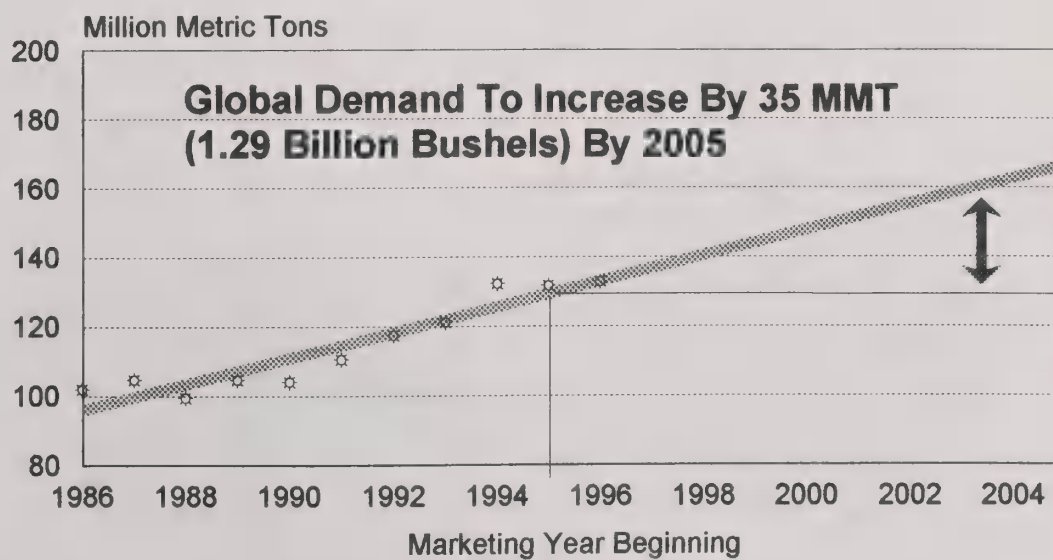
1987 - 1996



Source: USDA/NASS, November 1996

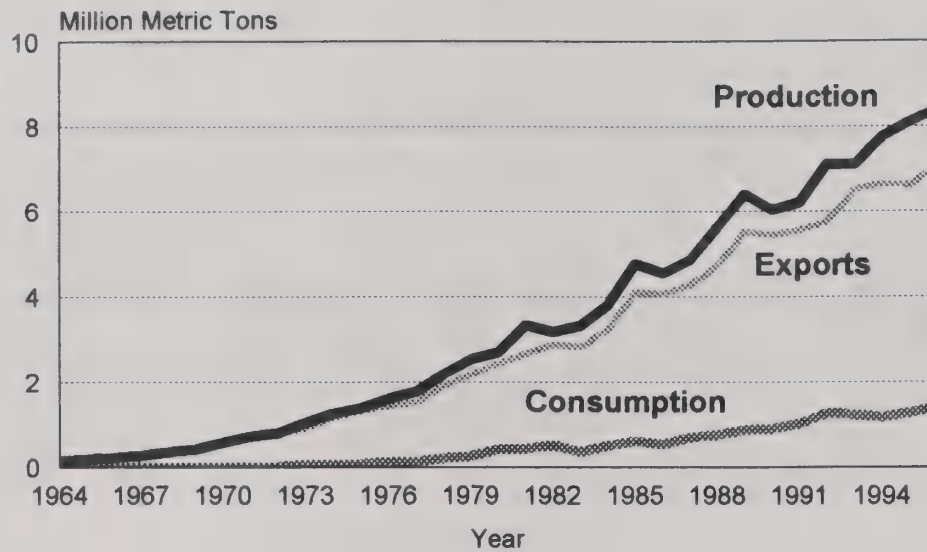
## Global Consumption of Soybeans

86 - 96 and Trend To 2005



## Malaysia

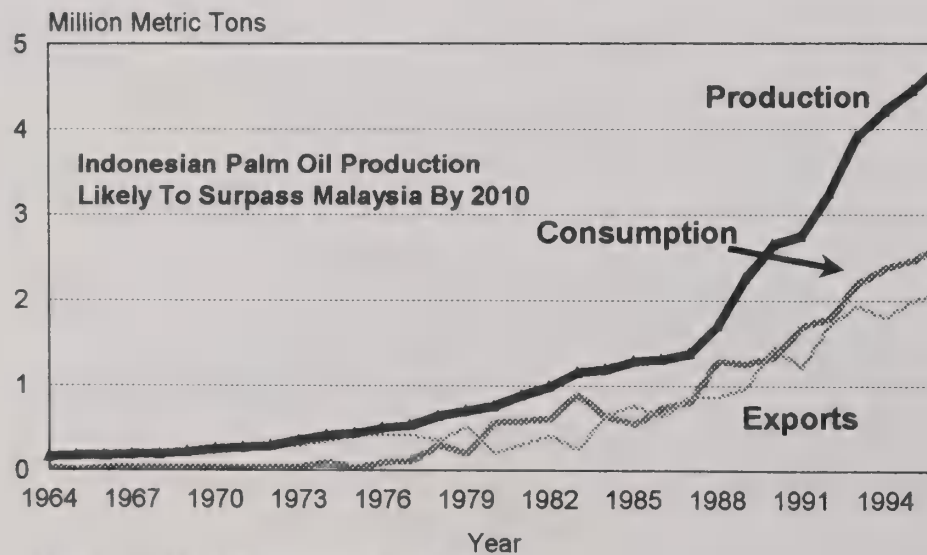
### Palm Oil Production, Consumption, and Exports



Source: USDA/NASS

## Indonesia

### Palm Oil Production, Consumption, and Exports

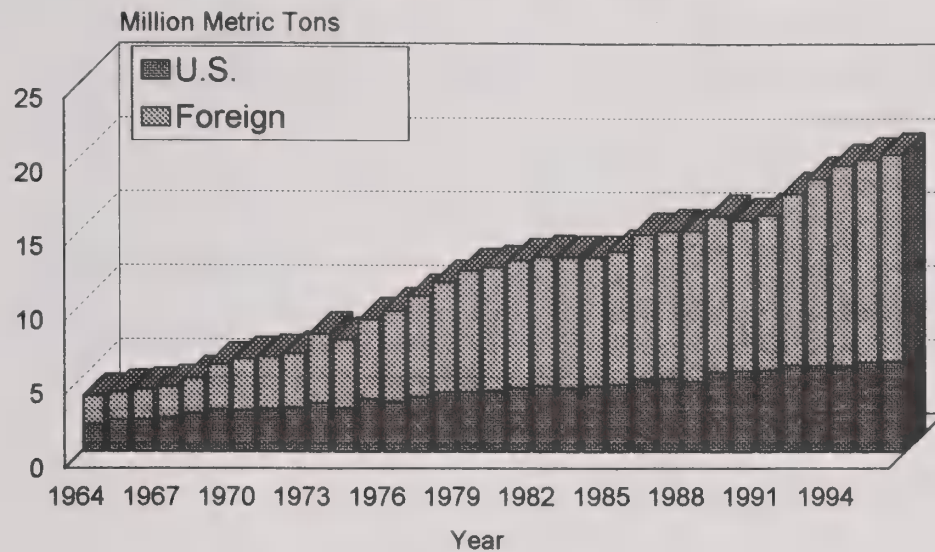


Source: USDA/NASS, September 1996



## U.S. and Foreign Consumption of Soybean Oil

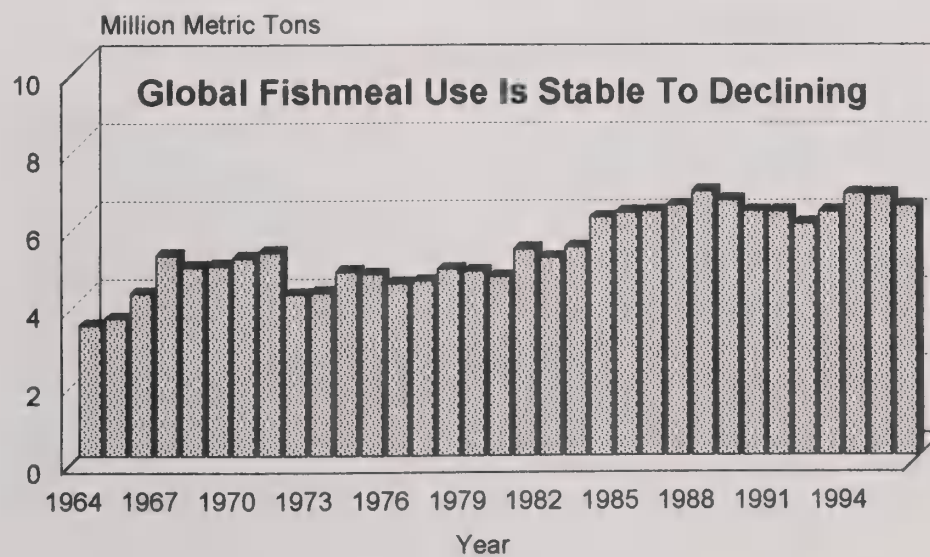
1964 - 1996



Source: USDA/NASS, September 1996

## Global Fishmeal Consumption

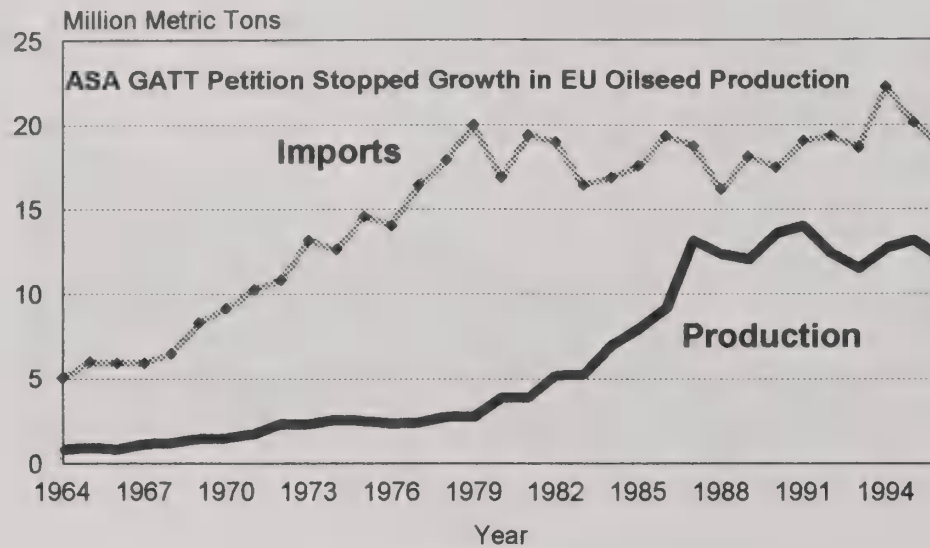
1964 - 1996



Source: USDA/NASS, September 1996

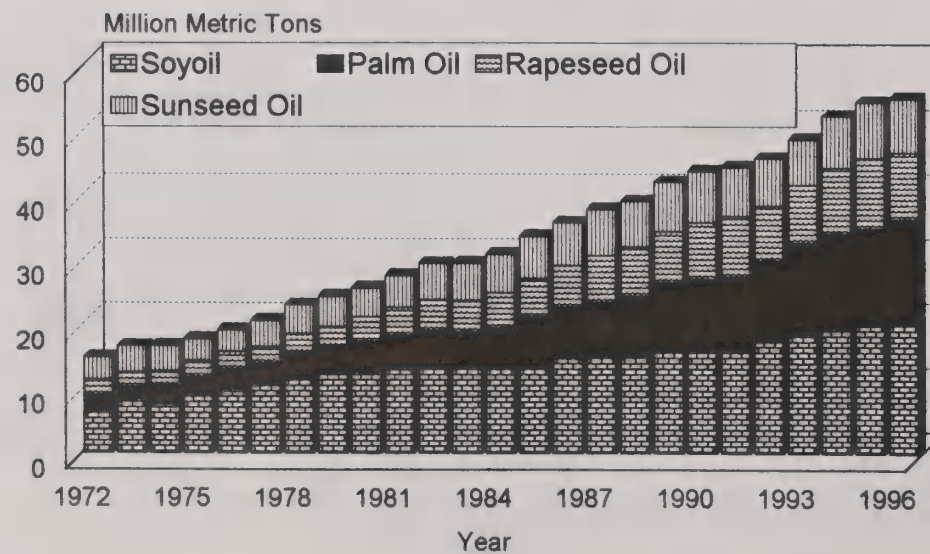
## European Union

### Production and Imports of Oilseeds 1964 - 1996



Source: USDA/NASS, September 1996

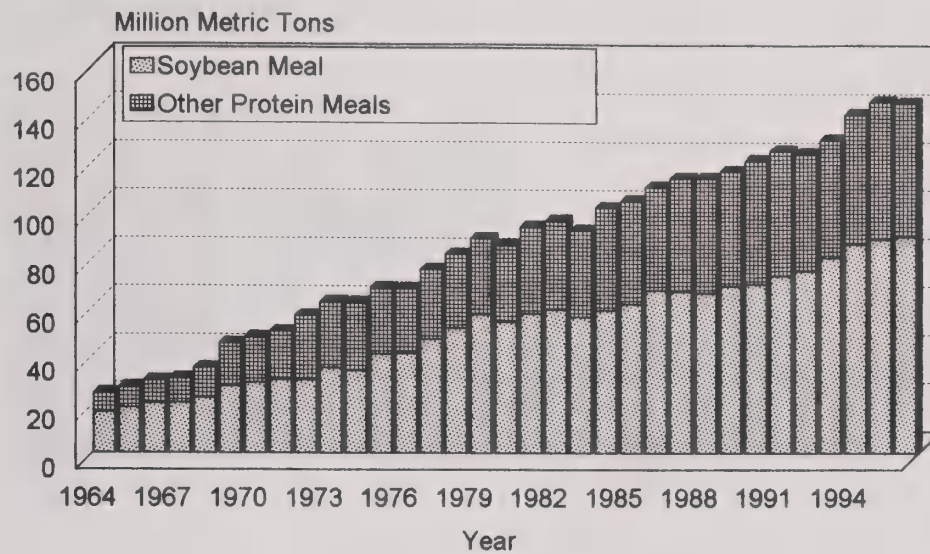
## Global Consumption of Soybean Oil, Rapeseed Oil, Palm Oil, and Sunseed Oil



Source: USDA/NASS, September 1996

## Global Protein Meal Consumption

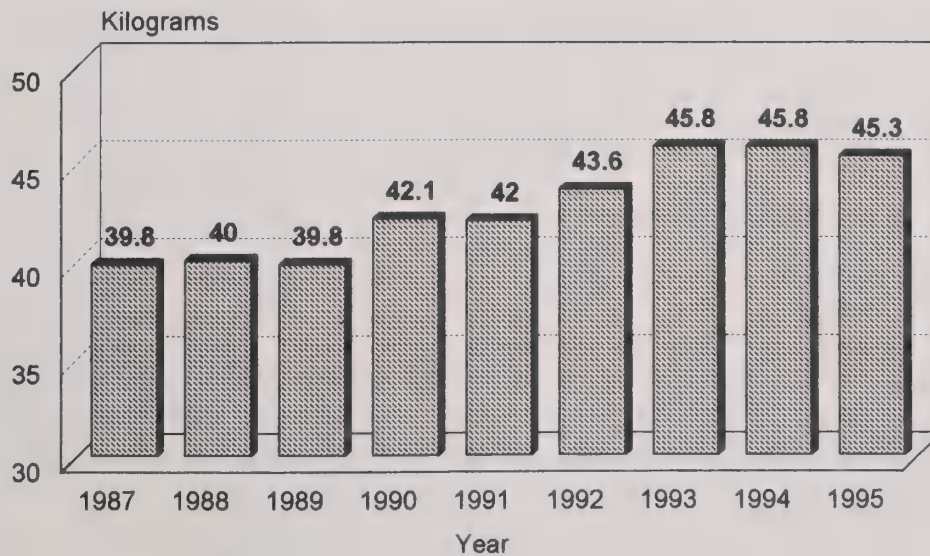
1964 - 1996



Source: USDA/NASS

## United States

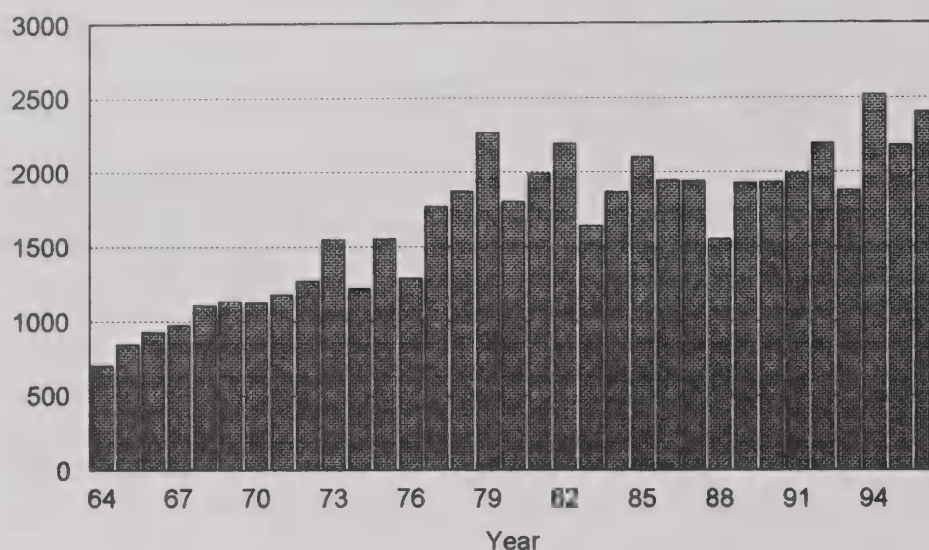
### Annual Per Capita Consumption of Fats and Oils



Source: Oil World

## U.S. Soybean Production

1964 - 1996



Source: USDA/NASS, November 1996

## U.S. Soybean Supply/Demand Estimates

1993/94 - 1996/97

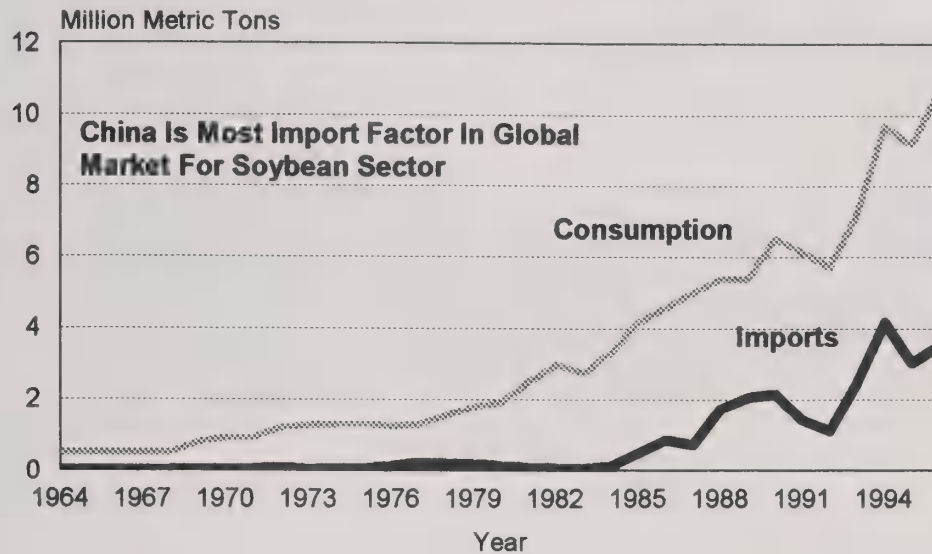
	1993/94	1994/95	1995/96	1996/97
Carry-In Stocks	292	209	335	183
Imports	6	5	4	4
Production	1871	<b>2517</b>	2177	<b>2382</b>
Domestic Crush	1276	<b>1405</b>	1370	1410
Domestic Use	1372	1558	1482	1525
Exports	589	838	851	<b>905</b>
Total Use	1961	<b>2396</b>	2333	2415
Ending Stocks	209	335	183	140

Source: USDA, February 1997



## China

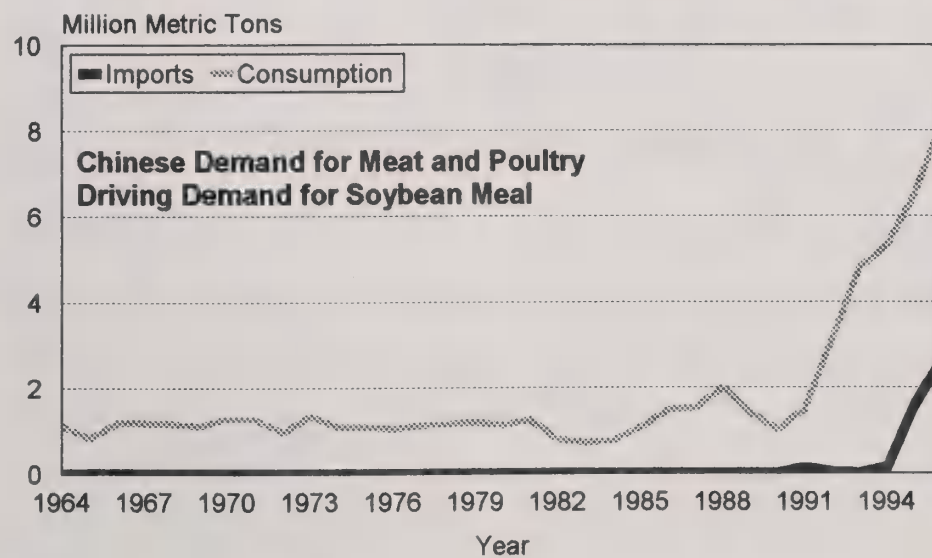
### Imports and Consumption of Vegetable Oils 1964 - 1996



Source: USDA/NASS, Oil World

## China

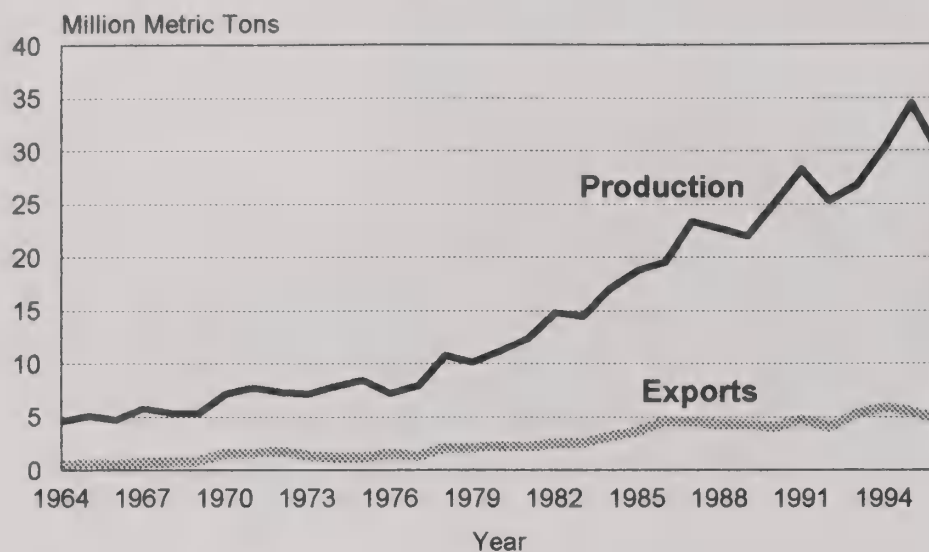
### Imports and Consumption of Soybean Meal 1964 - 1996



Source: USDA/NASS, Oil World

## Global Rapeseed Production and Exports

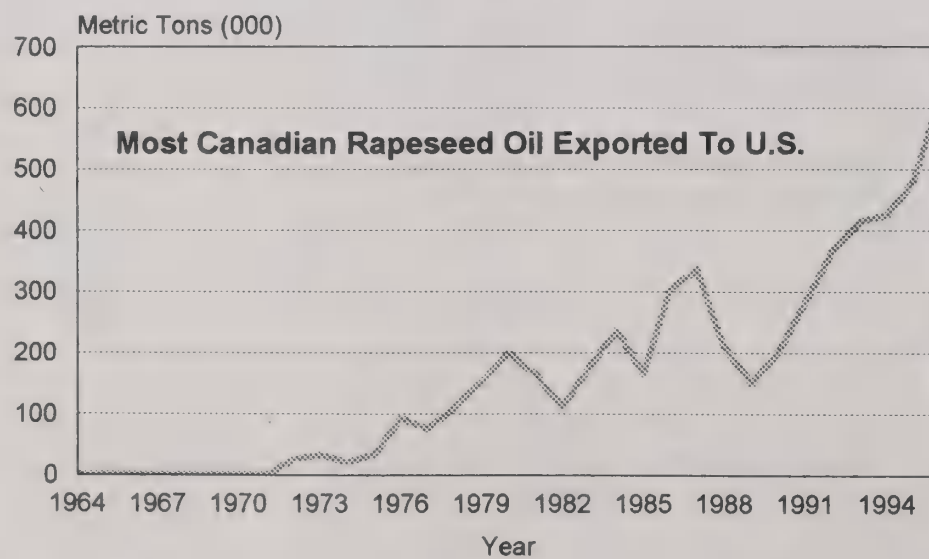
1964 - 1996



Source: USDA/NASS, September 1996

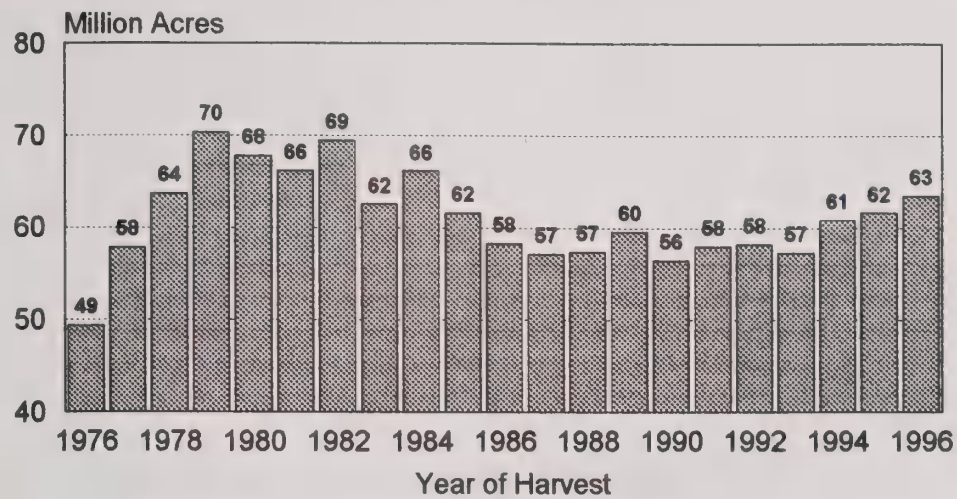
## Canadian Exports of Rapeseed Oil

1964 - 1996



Source: USDA/NASS, October 1996

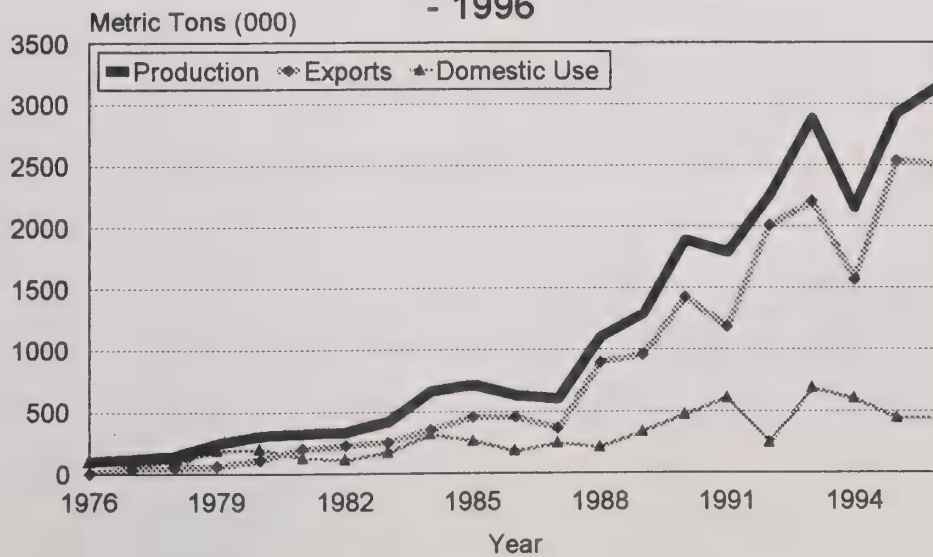
## U.S. Harvested Area of Soybeans 1976 - 1996



Source: USDA/NASS, November 1996

## India

### Production, Domestic Use and Exports of Soybean Meal 1976 - 1996



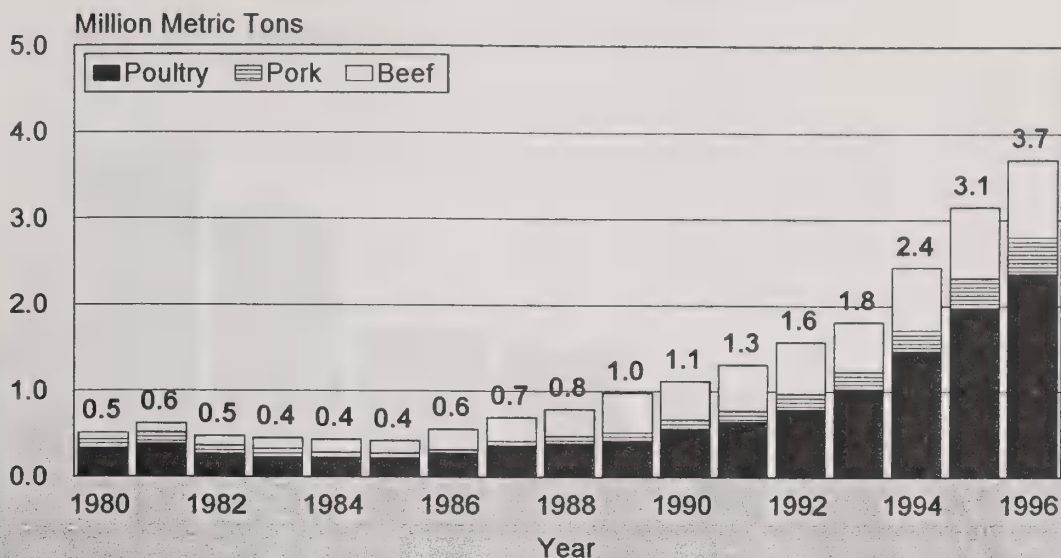
Source: USDA/NASS, Oil World





## U.S. Exports of Beef, Pork, and Poultry Meat

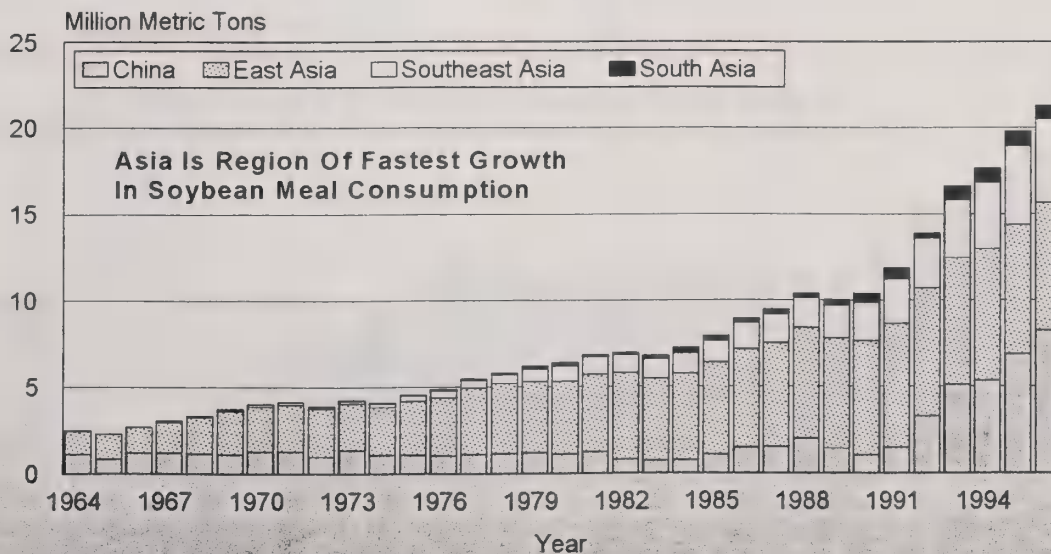
1980 - 1997



Source: USDA/NASS, December 1996

## Asian Soybean Meal Consumption

1964 - 1996

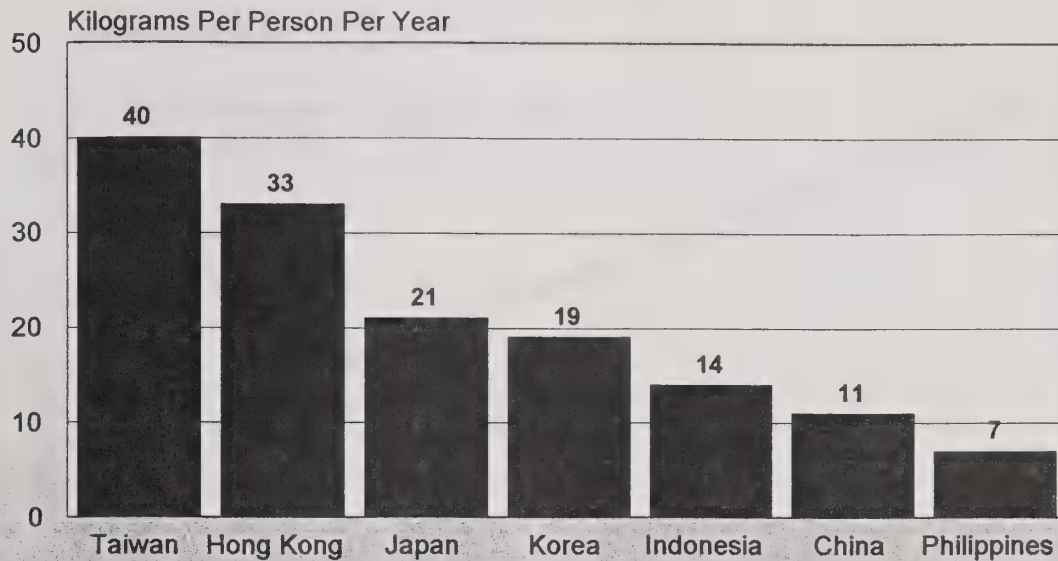


Source: USDA/NASS, February 1997



## Per Capita Vegetable Oil Consumption

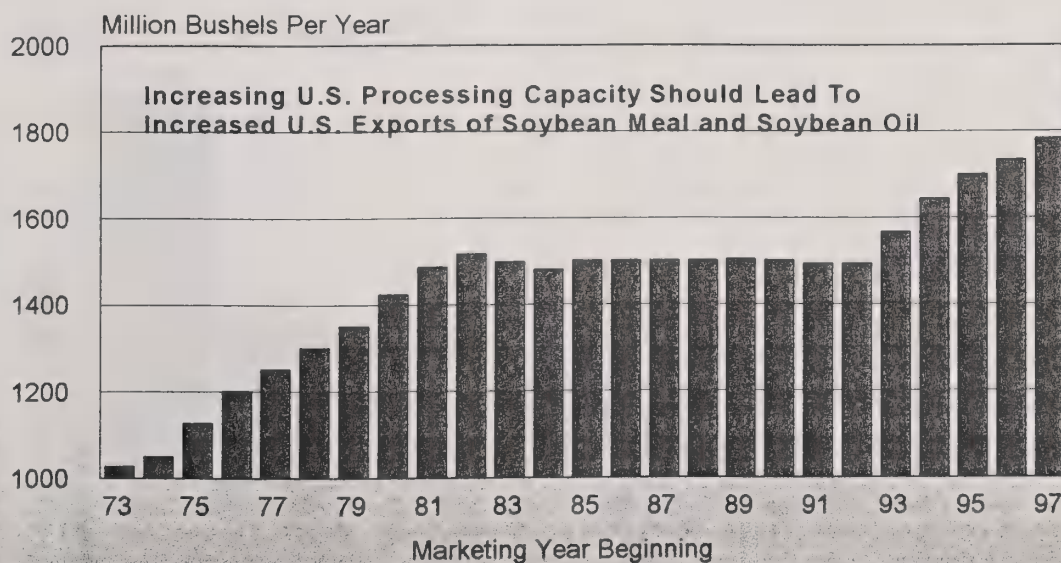
### Selected Asian Nations



Source: Oil World, 1996 Annual

## Estimated U.S. Soybean Processing Capacity

1973 - 1997

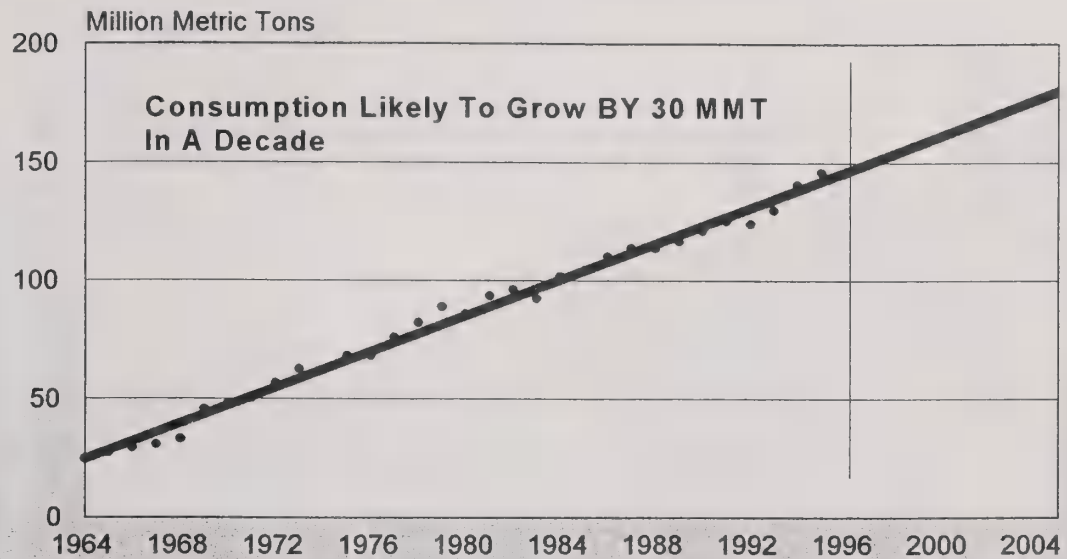






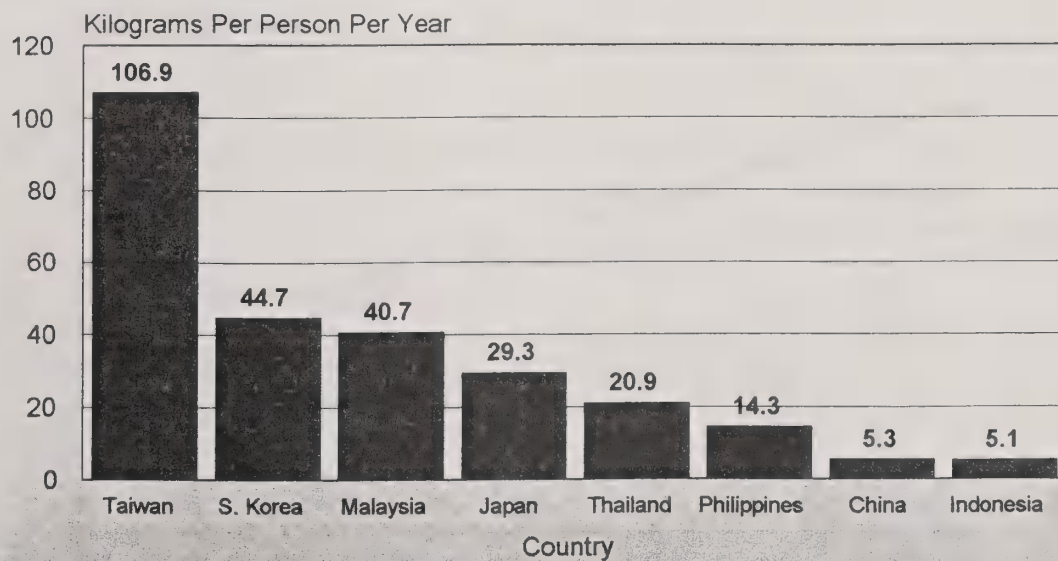
## Global Soybean Meal Consumption

1964 - 1996 and Trend to 2005



## Per Capita Soybean Meal Consumption

Selected Asian Nations

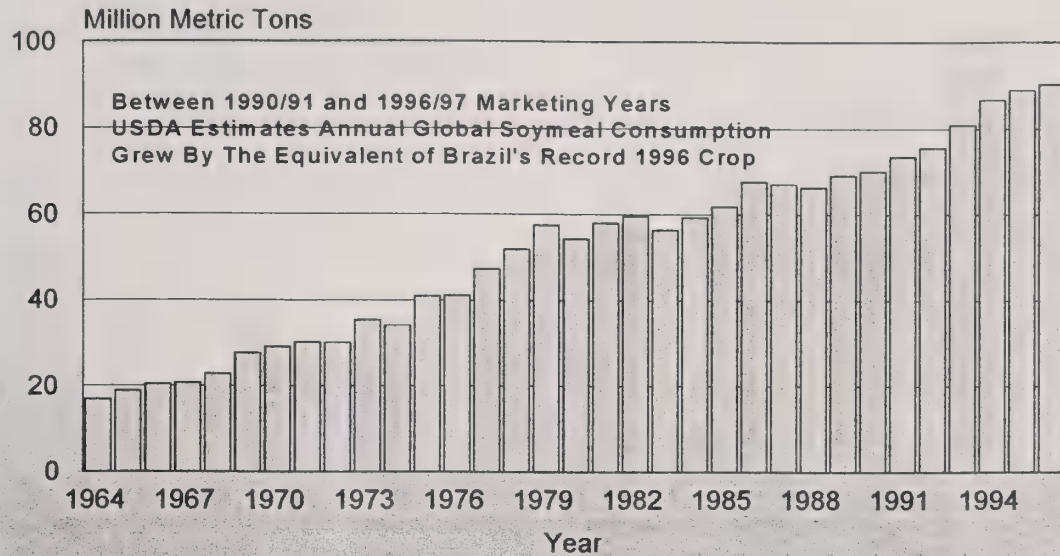


Source: Oil World



## Global Soybean Meal Consumption

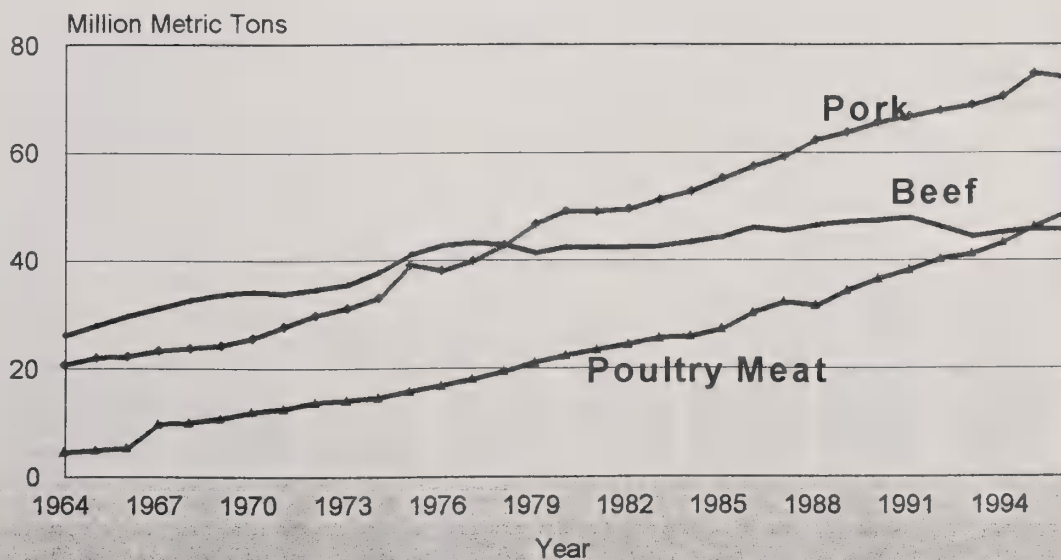
1964 - 1996



Source: USDA/NASS, February 1997

## Global Consumption of Beef, Pork, & Poultry

1964 - 1996



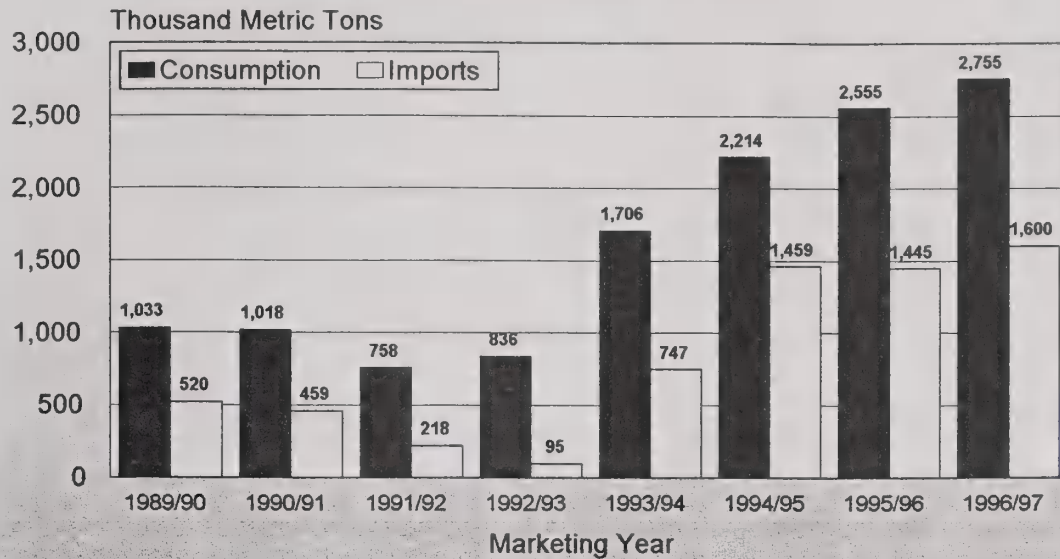
Source: USDA/NASS, February 1997





## China

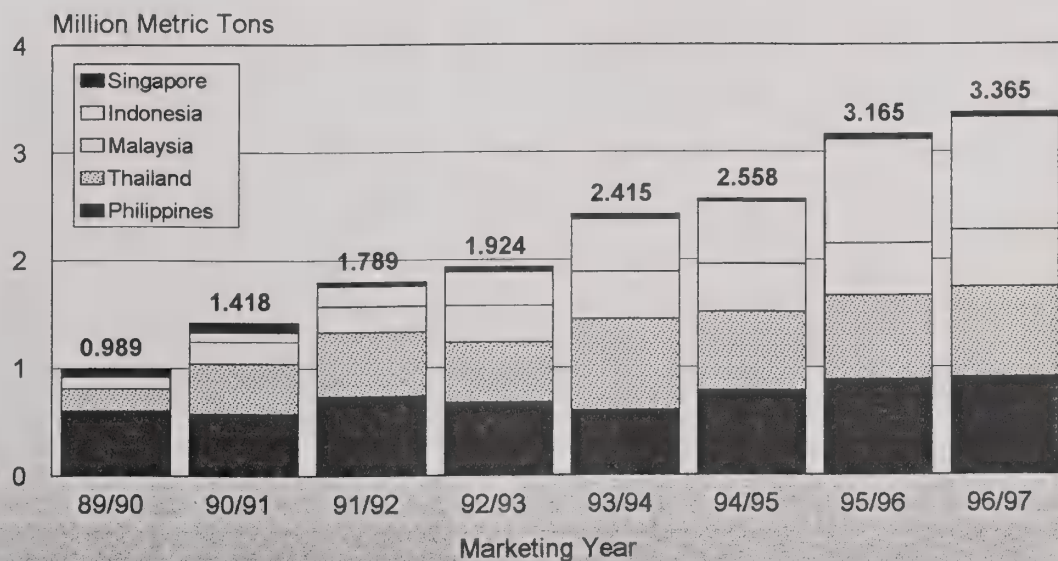
### Imports and Consumption of Soybean Oil



Source: Oil World, January 1997

## Southeast Asia

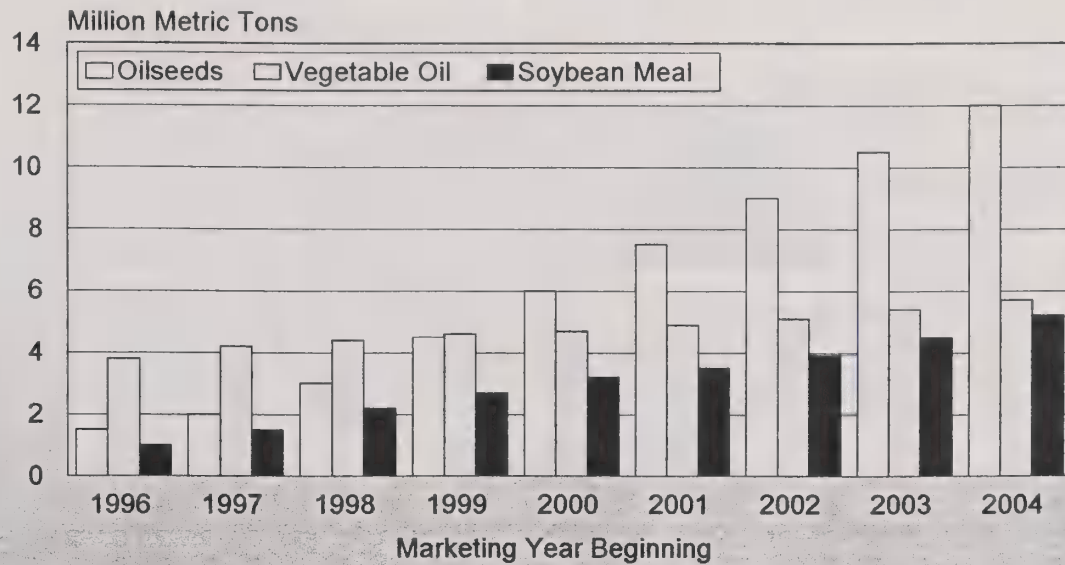
### Soybean Meal Imports



Source: Oil World



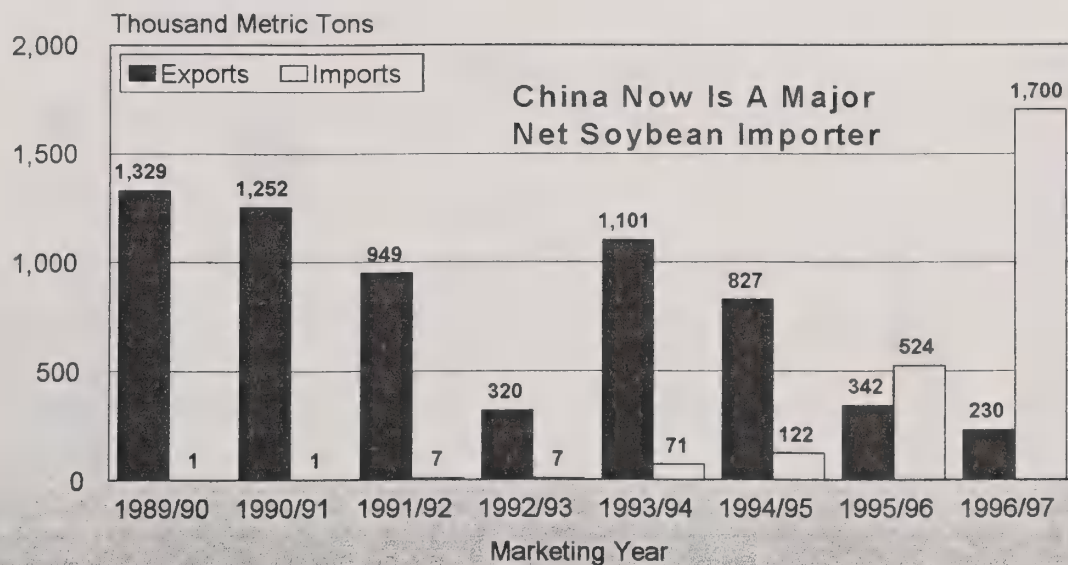
## Projected Chinese Import Needs Oilseeds, Vegetable Oils, & Soybean Meal



Source: China Working Group, American Oilseed Coalition

## China

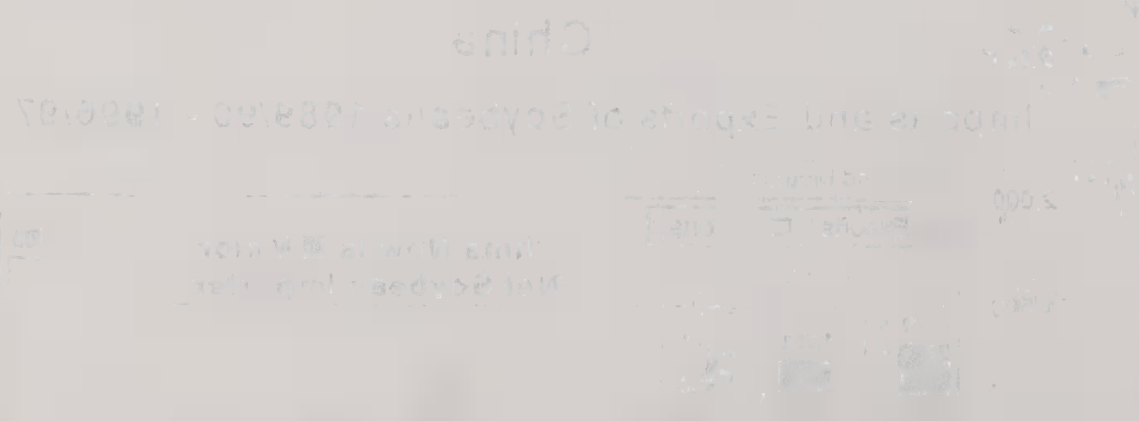
### Imports and Exports of Soybeans 1989/90 - 1996/97



Source: Oil World, January 1997

# Protected Chinese Import Needs Overseas Vegetables and Fruit Market

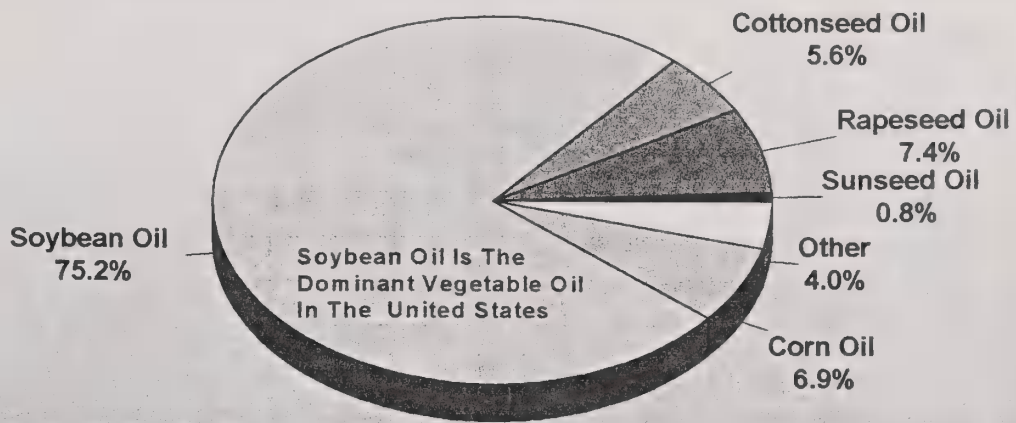
Table 1. Estimated Market Potential for Protected Chinese Imports in Overseas Markets, 1980-1990





# U.S. Vegetable Oil Consumption

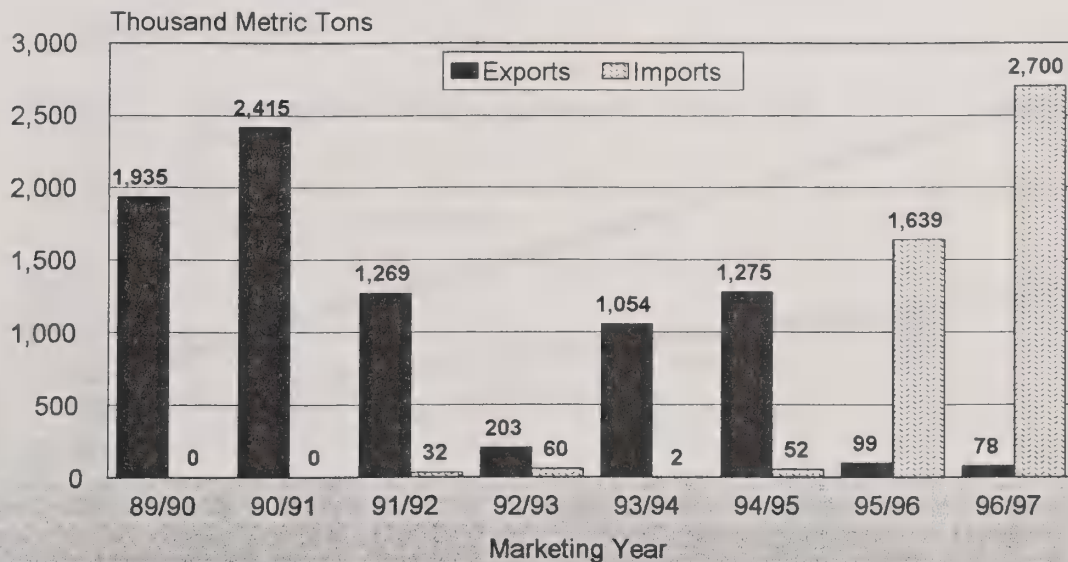
Market Share By Type of Oil in 1996/97



Source: Oil World, USDA/NASS

## China

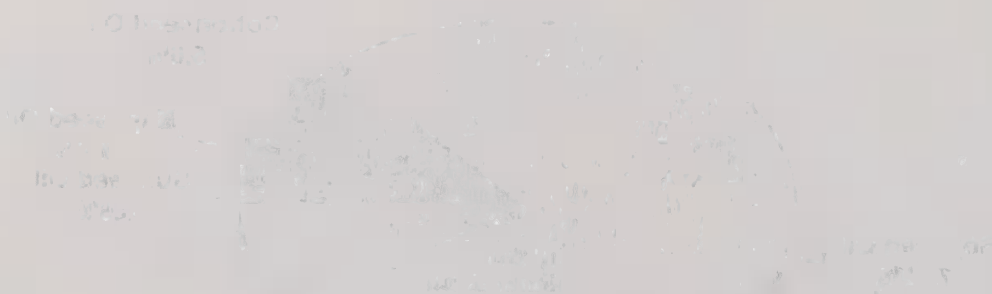
### Soybean Meal Exports and Imports



Source: Oil World

# U.S. Vegetable Oil Consumption

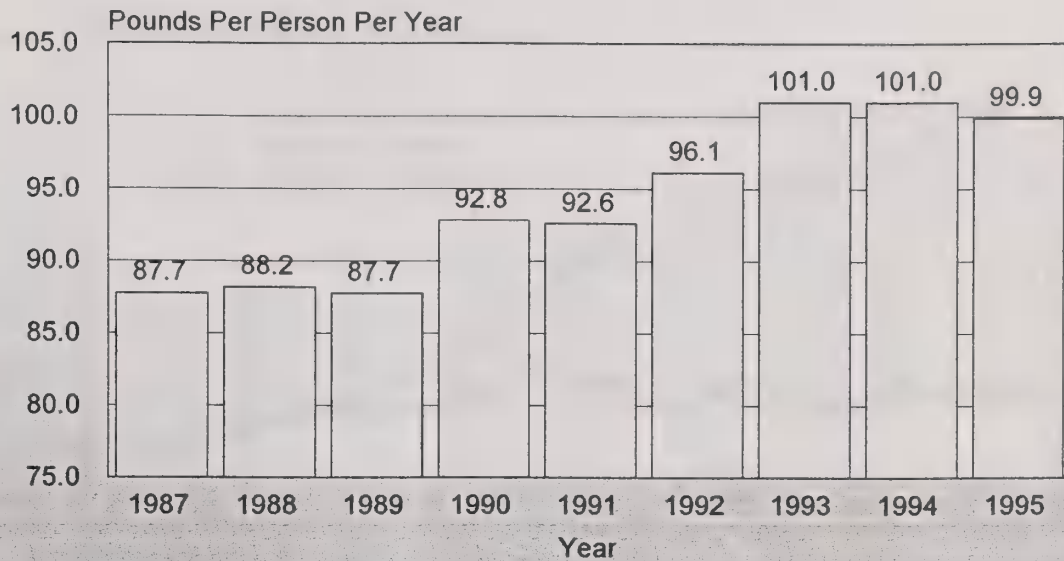
Market Size by Type of Oil in 1980



Source: U.S. Department of Agriculture, Economic Research Service, *Vegetable Oil Market*, Washington, D.C., 1981.

## United States

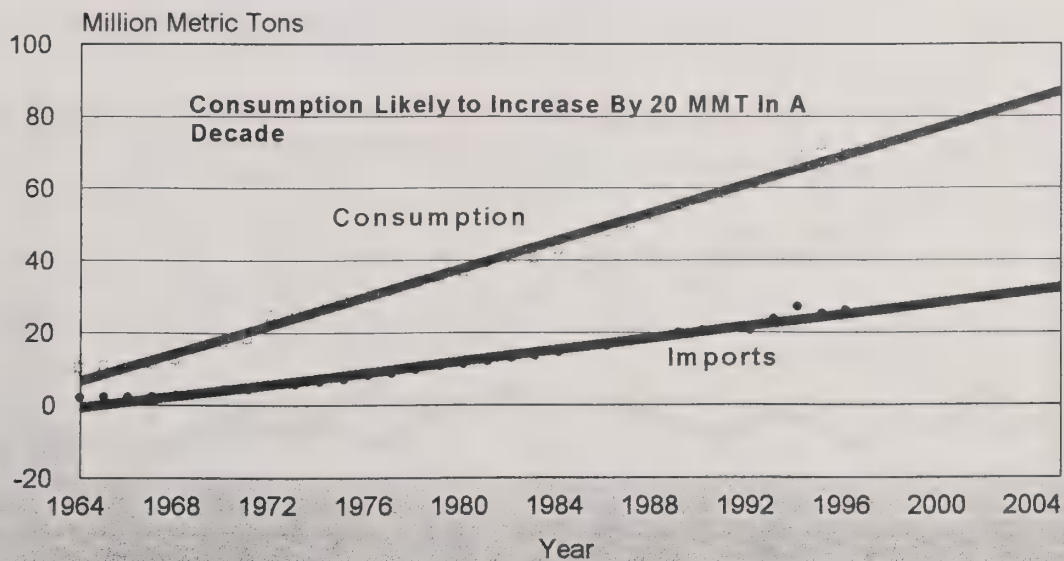
### Annual Per Capita Consumption of Fats and Oils



Source: Oil World

## Global Vegetable Oil Imports and Consumption

1964 - 1996 and Trend to 2005

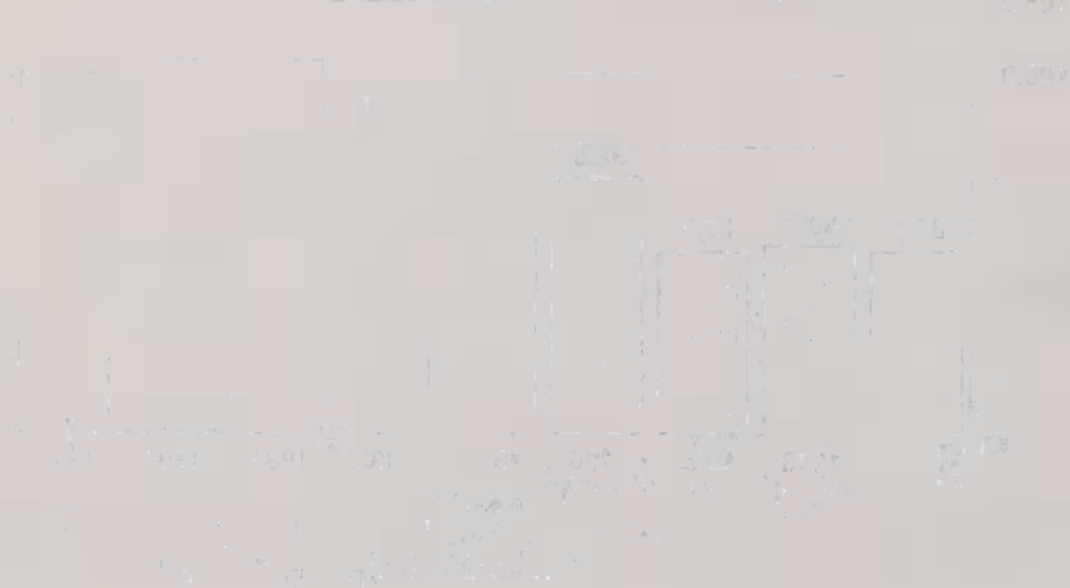


Source: USDA/NASS

# United States

Annual Net Consumption of Oil and Gas

Thousands of Barrels per Year



# Global Vegetable Oil Imports and Consumption

1984 - 1990 and Trends to 2000

Million Metric Tons

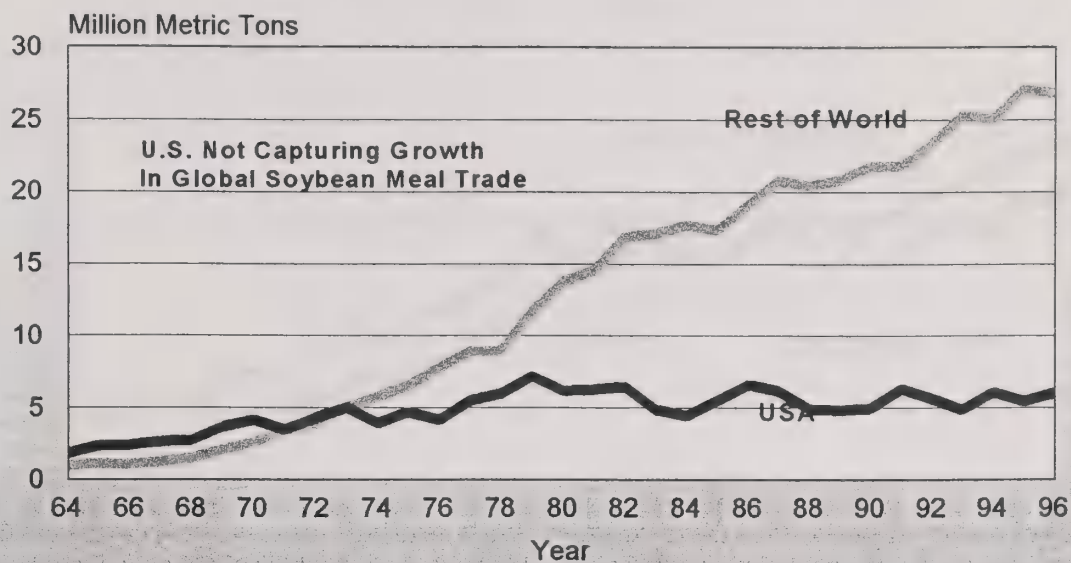
Consumption (Solid line) and Imports (Dashed line)





## Soybean Meal Exports

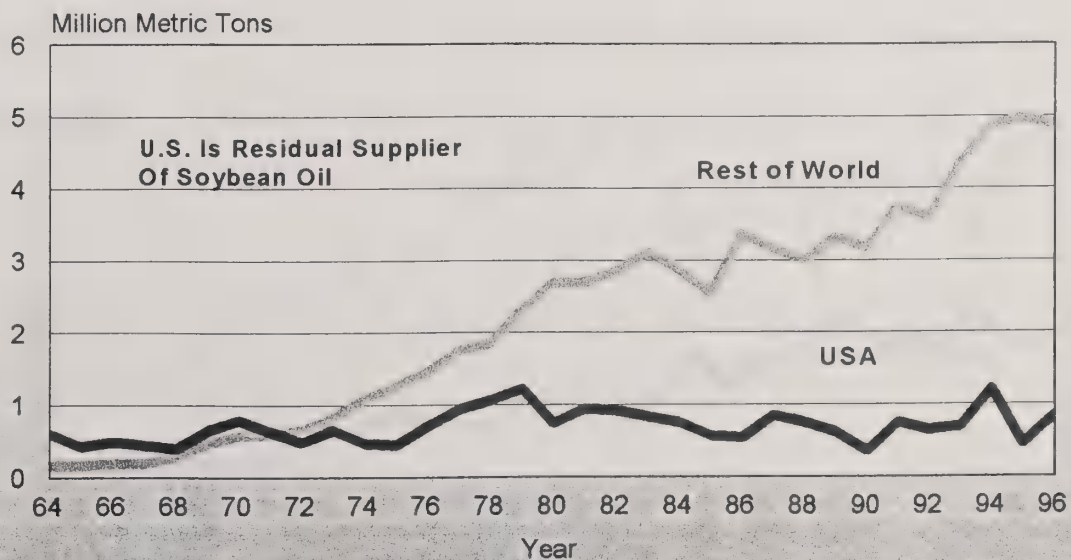
### U.S. Exports Versus Rest of World



Source: USDA/NASS, February 1996

## Soybean Oil Exports

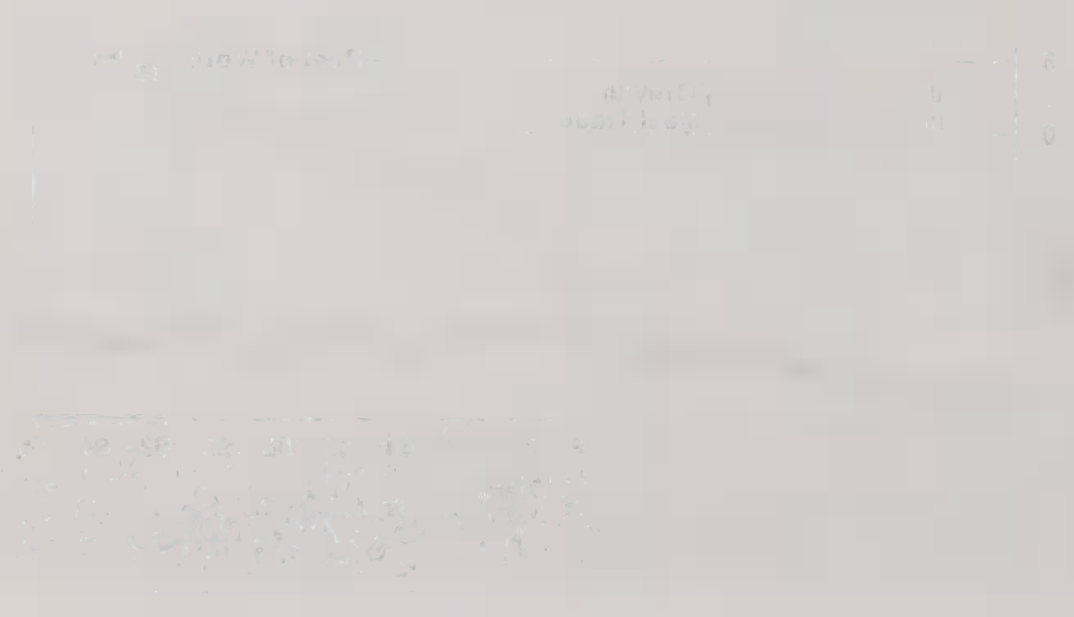
### U.S. Versus Rest Of World



Source: USDA/NASS, February 1997

# Soybean Meal Exports U.S. Exports Versus Rest of World

Year - Million Tons



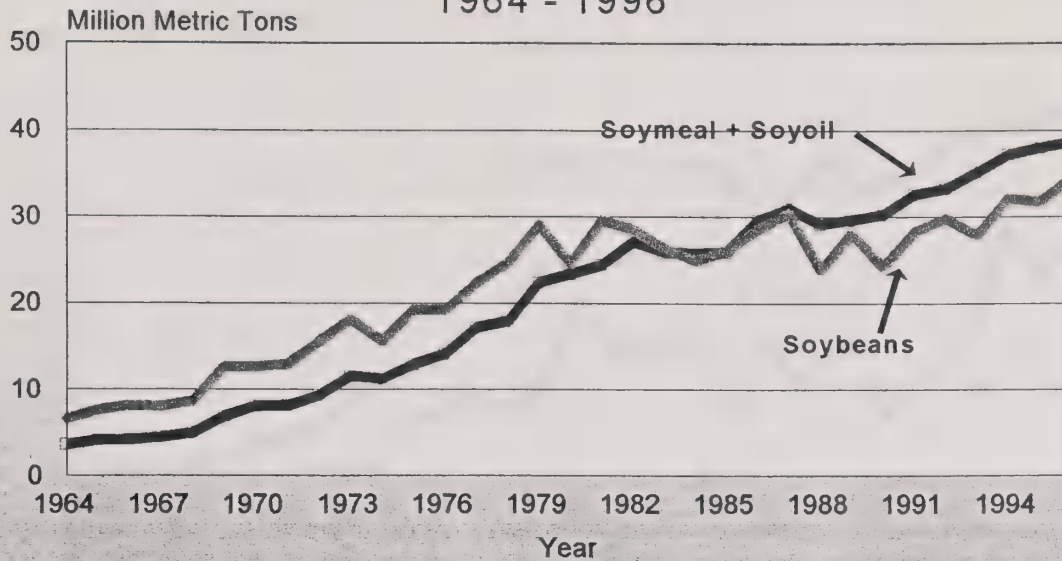
## Soybean Oil Exports

Year - Million Tons



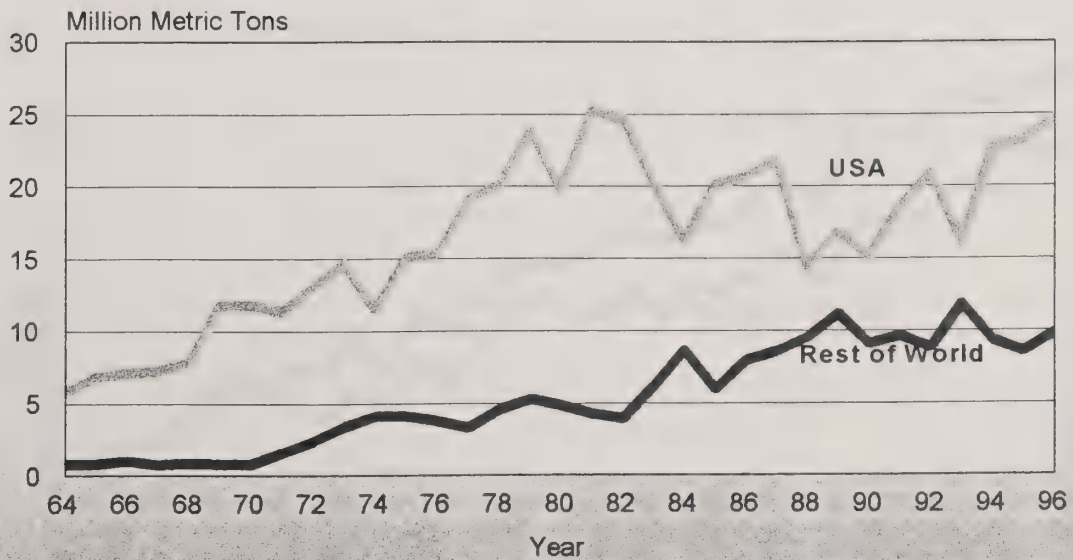
## Global Exports of Soybeans Versus Exports of Soybean Meal and Soybean Oil

1964 - 1996



Source: USDA/NASS, February 1997

## Soybean Exports U.S. Versus Rest of World



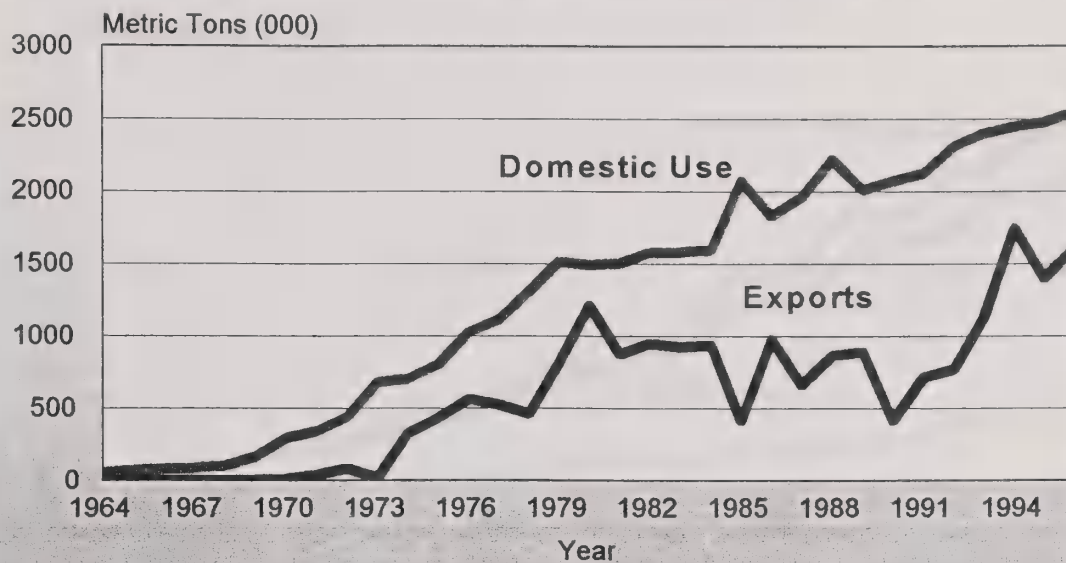
Source: USDA/NASS, February 1995





## Brazil

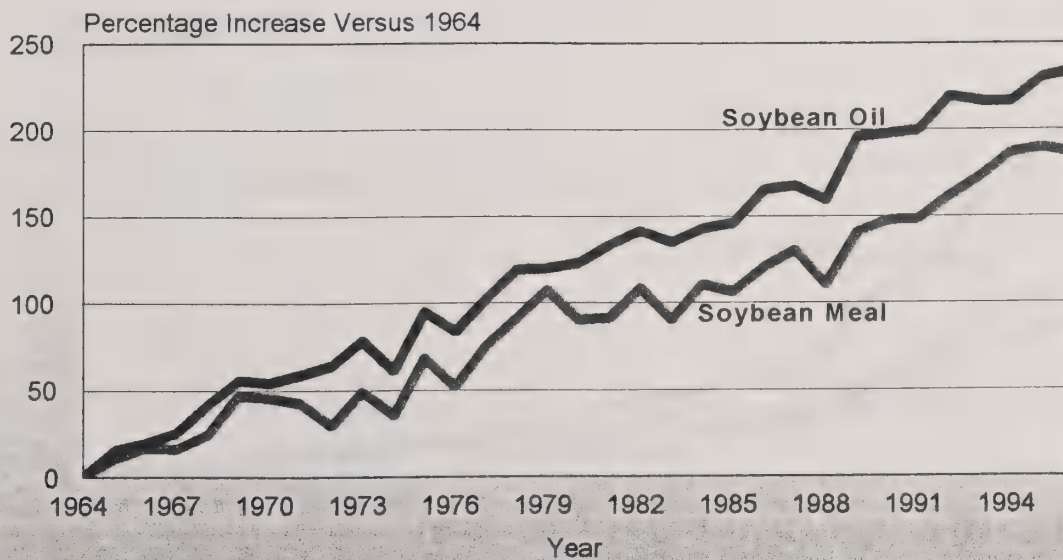
### Exports and Domestic Use of Soybean Oil 1964 - 1996



Source: USDA/NASS, February 1997

## U.S. Growth In Domestic Consumption

### Soybean Meal and Soybean Oil 1964 - 1996

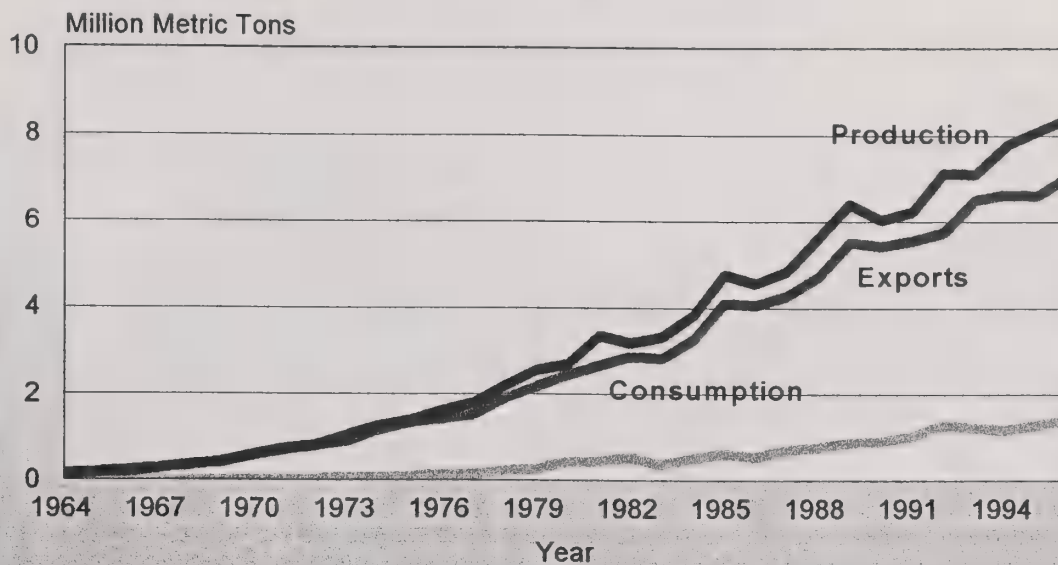


Source: USDA/NASS, November 1996



## Malaysia

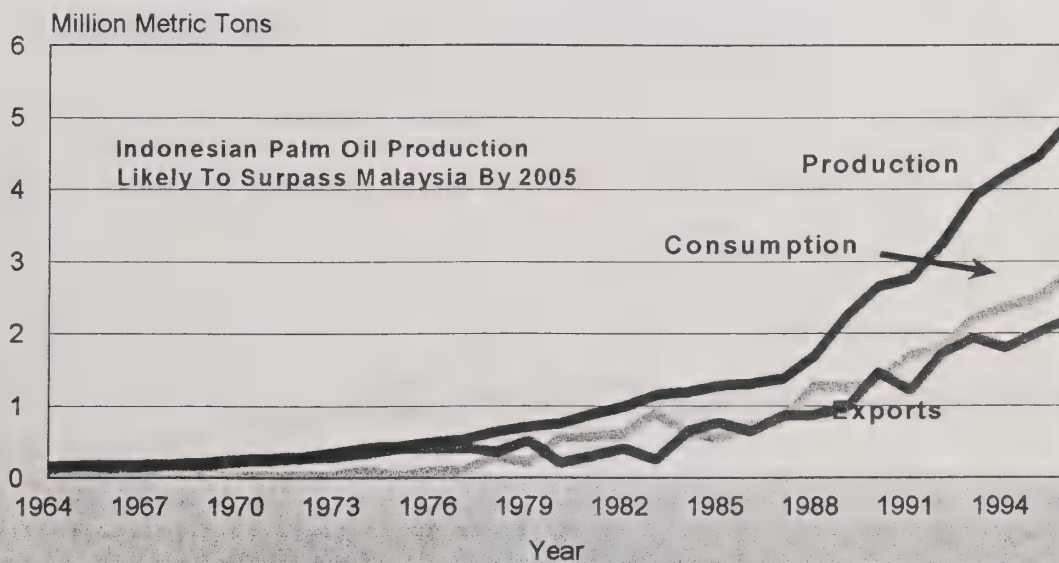
### Palm Oil Production, Consumption, and Exports



Source: USDA/NASS

## Indonesia

### Palm Oil Production, Consumption, and Exports



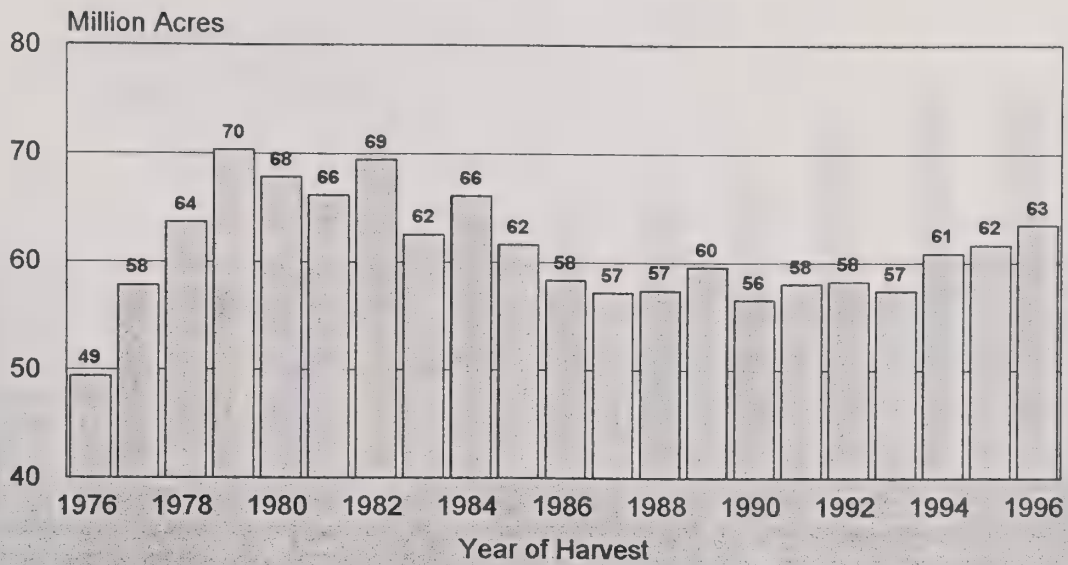
Source: USDA/NASS, February 1997





## U.S. Harvested Area of Soybeans

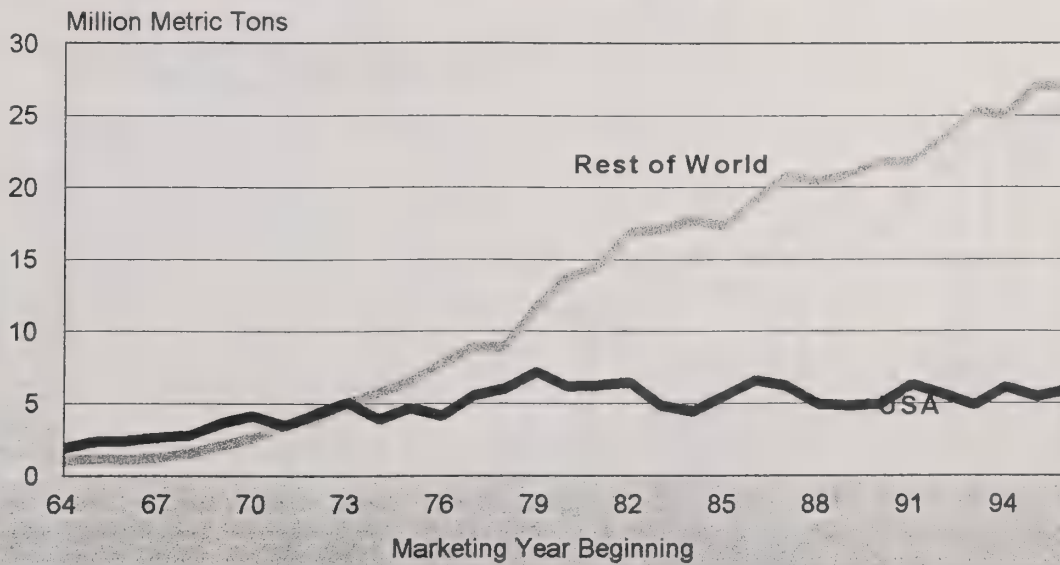
1976 - 1996



Source: USDA/NASS, February 1997

## Soybean Meal

U.S. Exports Versus Exports By Rest of World

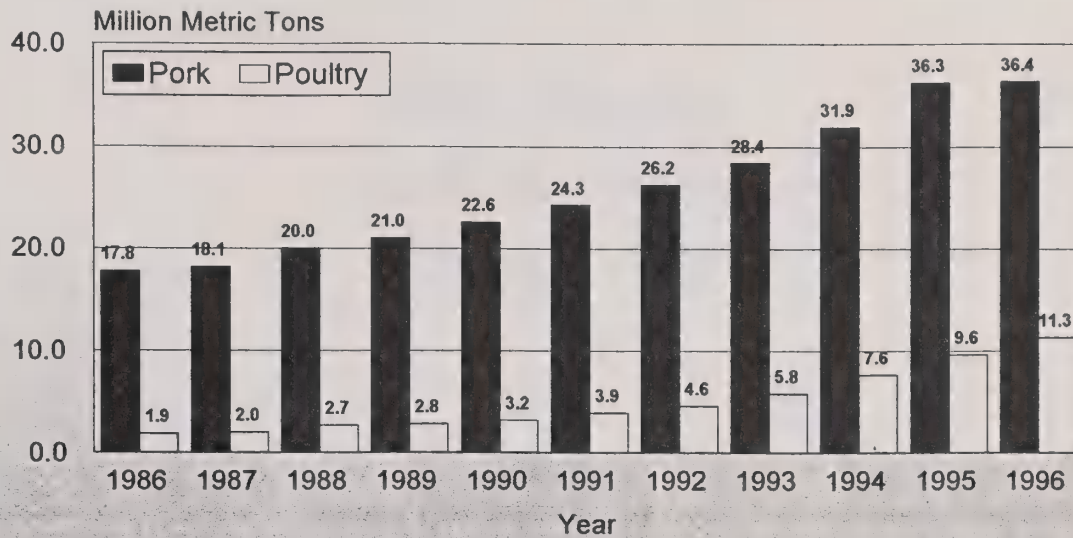


Source: USDA/NASS, February 1997



# China

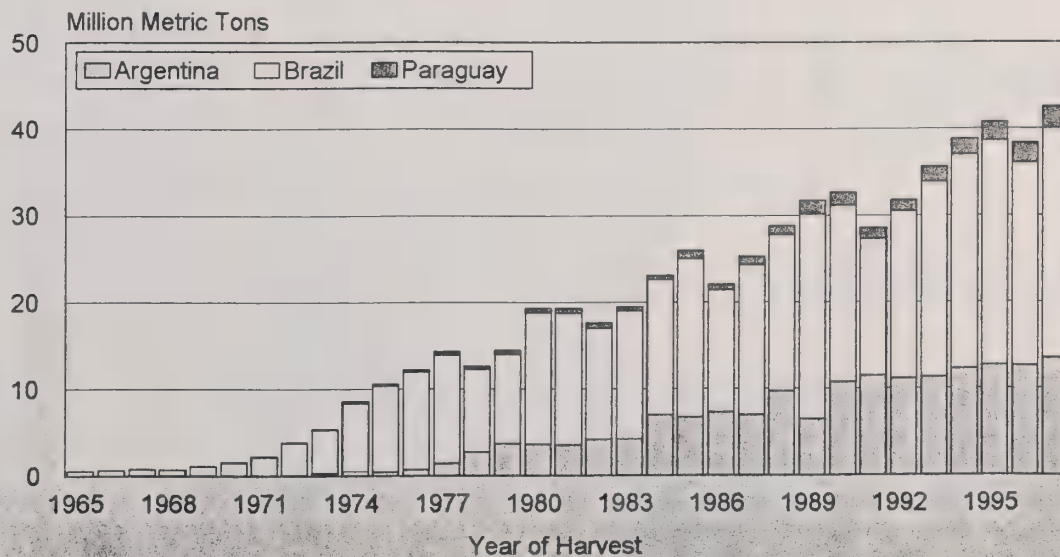
## Consumption of Pork and Poultry



Source: USDA/NASS

# South American Soybean Production

1965 - 1997



Source: USDA/NASS, February 1997

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*Benchmarking to Achieve World-Class  
Performance*

**Bill Windle**  
A.T. Kearney  
Chicago, IL

# 46th Oilseed Conference

**Processing Efficiency:  
Meeting the Challenge**

**March 9–11, 1997**

Hotel Monteleone

New Orleans, Louisiana, USA

Co-Sponsored by:

The American Oil Chemists' Society (AOCS)

The National Cottonseed  
Products Association, Inc.

Southern Regional Research  
Center/ARS/USDA







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**46th Oilseed Conference  
New Orleans, Louisiana**

**Benchmarking to Achieve  
World-Class Performance**

**March 10, 1997**

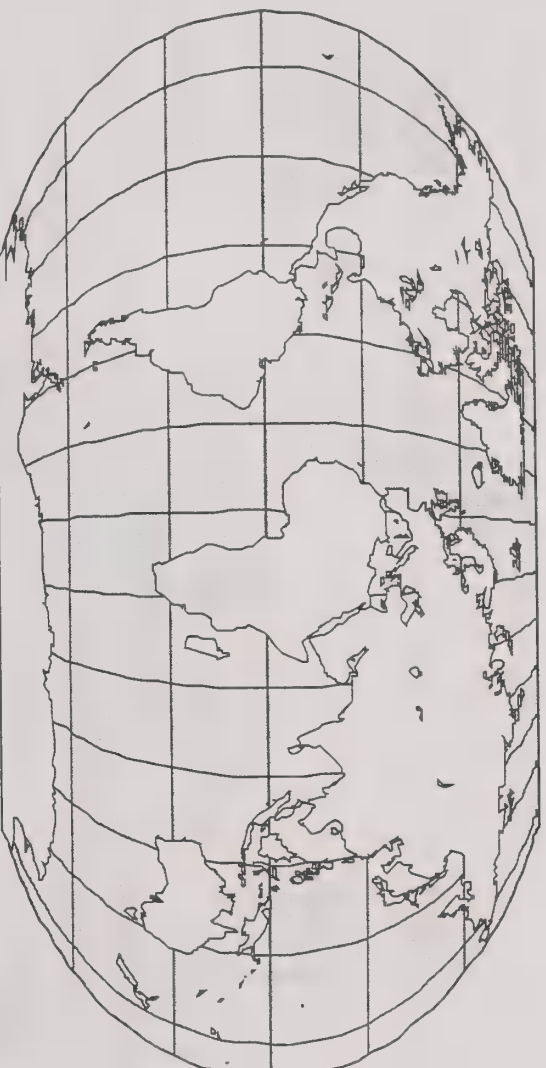
# Agenda

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- Introduction
- What is benchmarking?
- World-class supply chain performance
- Our future challenge



# A.T. Kearney — who we are



- Diversified management consulting firm
- Founded 1926
- 2,000 engagements per year
- CAGR > 25% since 1980

Alexandria  
Amsterdam  
Atlanta  
Barcelona  
Beijing  
Berlin  
Boston  
Brussels  
Buenos Aires  
Cambridge  
Caracas

Chicago  
Cleveland  
Copenhagen  
Coraopolis  
Dallas  
Denver  
Düsseldorf  
Englewood  
Helsinki  
Hong Kong

Houston  
Kuala Lumpur  
La Défense  
Lisbon  
London  
Los Angeles  
Madrid  
Manila  
Melbourne  
Mexico City

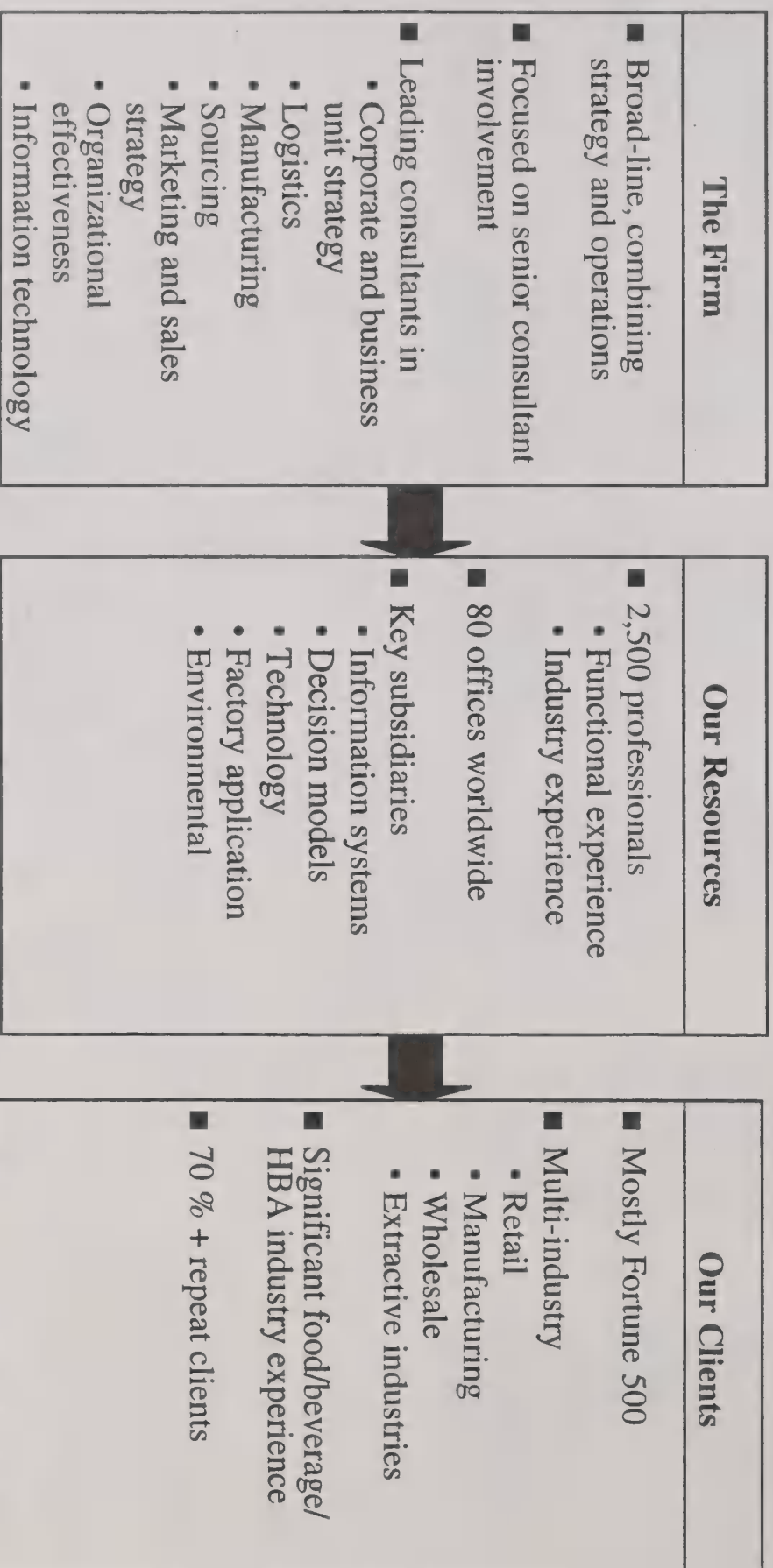
Miami  
Milan  
Minneapolis  
Moscow  
Munich  
New York  
Oslo  
Ottawa  
Paris  
Phoenix

Plano  
Prague  
Roslyn  
San Diego  
San Francisco  
Santa Clara  
São Paulo  
Seoul  
Singapore  
Southfield

St. Louis  
Stamford  
Stockholm  
Stuttgart  
Sydney  
Tokyo  
Toronto  
Warsaw  
Wellington  
Zug

**Leading the way in growth, quality,  
and achieving tangible results**

# A.T. Kearney — who we are

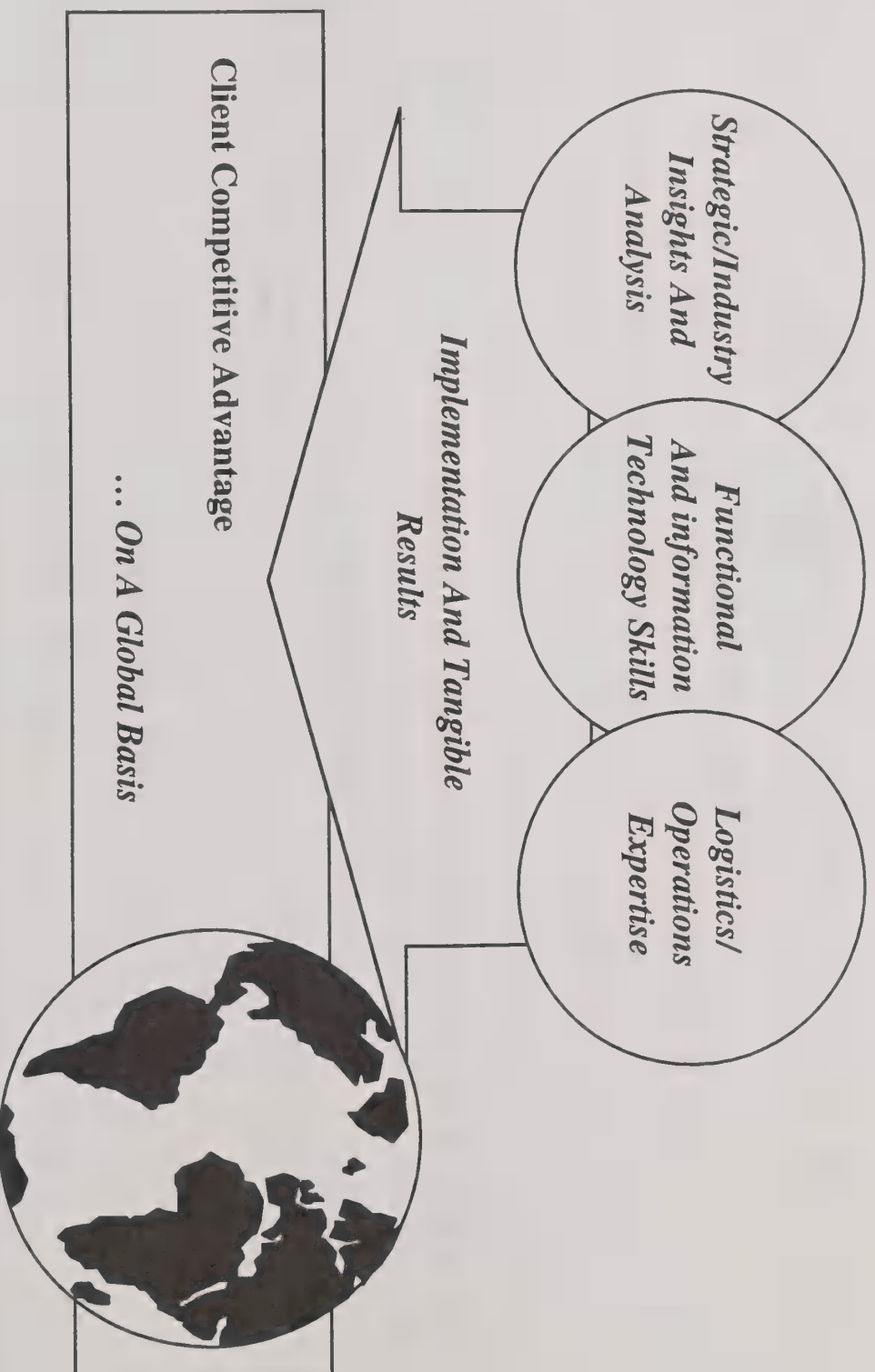


**Supported by the leading-edge technology resources of EDS**

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# Our strengths

- One of the very few consulting firms that offers a combination of ...



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## Benchmarking — what is it?

<b>Benchmark</b>
------------------

A point of reference from which measurements may be made. Something that serves as a standard by which others may be measured — <i>Webster's</i>
--

<b>Benchmarking</b>
---------------------

An objective and comparative evaluation of functions, processes, structures, cost and performance using indicators established through direct research among a representative group of similar or competing organizations
---

— *A.T. Kearney*



---

# Benchmarking — why is it valuable?

- Benchmarking measures and amplifies competitive and organizational differences
- Benchmarking focuses an organization's attention on competitive-driven necessities
  - Doing the right things
  - Doing things right
  - Organizing to win
- Benchmarking provides a leadership target

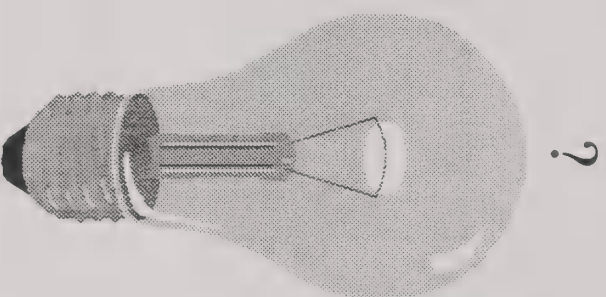
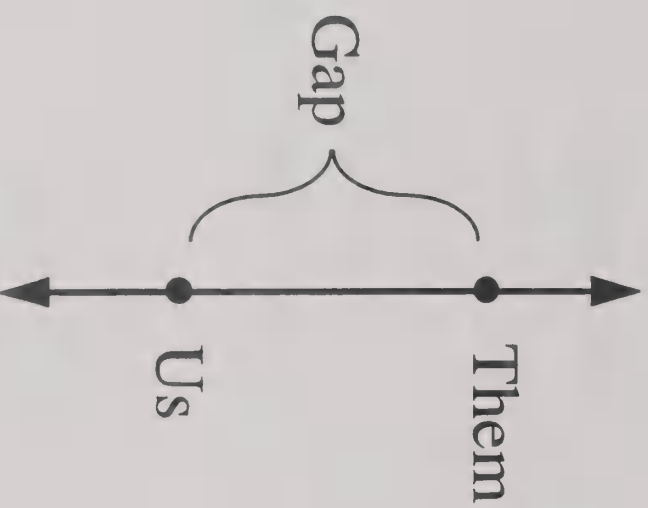
**“The Best Of The Best”**

# Benchmarking — why is it valuable?

**Competitive  
Comparison**

**Breakthrough  
Ideas**

**Credibility  
For Change**



We Can't... But  
Others Are

# Benchmarking — why is it valuable?

Without Benchmarking	With Benchmarking
<ul style="list-style-type: none"> <li>• Becoming competitive               <ul style="list-style-type: none"> <li>— Internally focused</li> <li>— Evolutionary change</li> <li>— Low commitment</li> </ul> </li> </ul>	<ul style="list-style-type: none"> <li>• Concrete understanding of competition</li> <li>• New ideas of proven practices and technology</li> <li>• High commitment</li> </ul>
<ul style="list-style-type: none"> <li>• Developing true measures of productivity               <ul style="list-style-type: none"> <li>— Pursuing pet projects</li> <li>— Strengths and weaknesses not understood</li> <li>— Route of least resistance</li> </ul> </li> </ul>	<ul style="list-style-type: none"> <li>• Solving real problems</li> <li>• Understanding outputs</li> <li>• Based on industry best practices</li> </ul>
<ul style="list-style-type: none"> <li>• Industry best practices               <ul style="list-style-type: none"> <li>— Not invented here</li> <li>— Few solutions</li> <li>— Average of industry progress</li> <li>— Frantic catch-up activity</li> </ul> </li> </ul>	<ul style="list-style-type: none"> <li>• Proactive search for change</li> <li>• Many options</li> <li>• Business practice breakthrough</li> <li>• Superior performance</li> </ul>
<ul style="list-style-type: none"> <li>• Defining customer requirements               <ul style="list-style-type: none"> <li>— Based on history or gut feel</li> <li>— Perception</li> <li>— Low fit</li> </ul> </li> </ul>	<ul style="list-style-type: none"> <li>• Market reality</li> <li>• Objective evaluation</li> <li>• High conformance</li> </ul>
<ul style="list-style-type: none"> <li>• Establishing effective goals and objectives               <ul style="list-style-type: none"> <li>— Lacking external focus</li> <li>— Reactive</li> <li>— Lagging industry</li> </ul> </li> </ul>	<ul style="list-style-type: none"> <li>• Credible, unarguable</li> <li>• Proactive</li> <li>• Industry leading</li> </ul>

# Benchmarking — a word of caution

## Benchmarking Is

- A valuable tool for comparisons
- A logical step for enhancing performance measurement and target setting

## Benchmarking Is Not

- All the answers
- Without conflict and is subject to interpretation

## Benchmarking Should

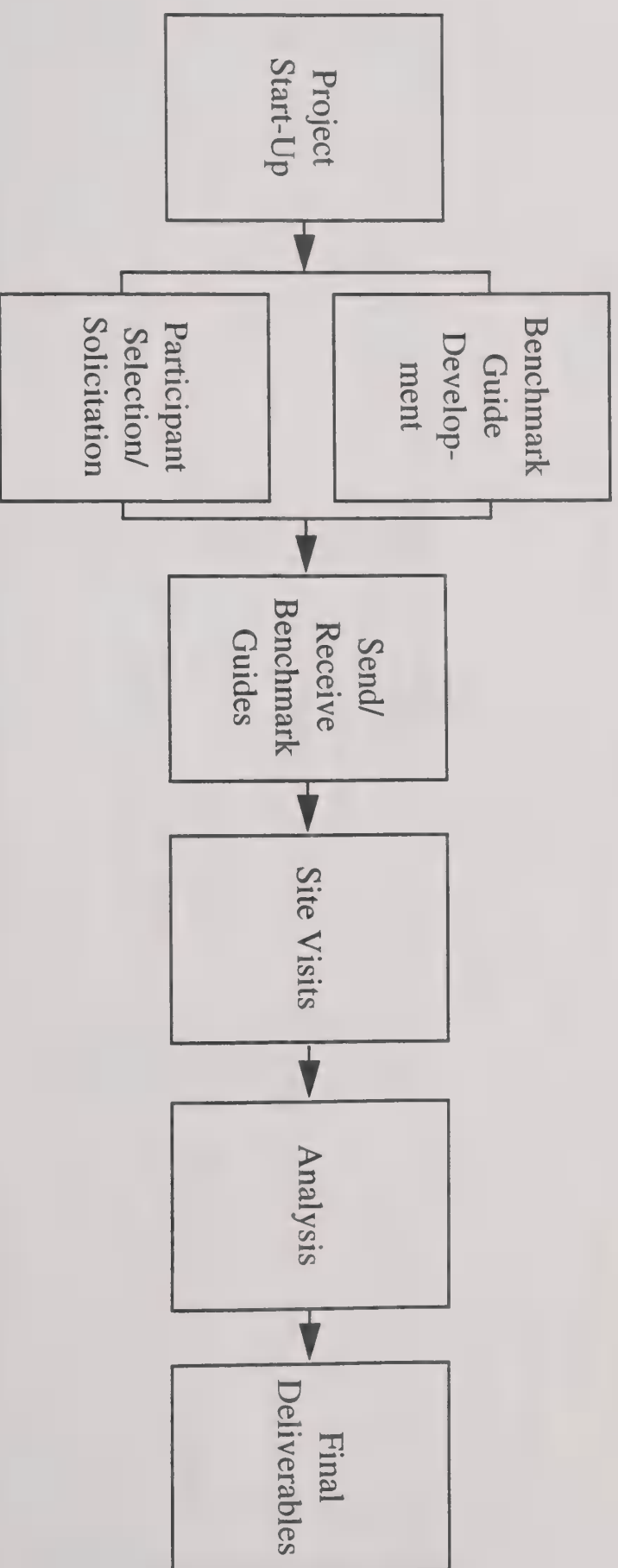
- Be used judiciously
- Have indices analyzed in sets
- Use trends rather than just absolutes



**Benchmarking — a first step in creating the recognition that change and improvement is needed**

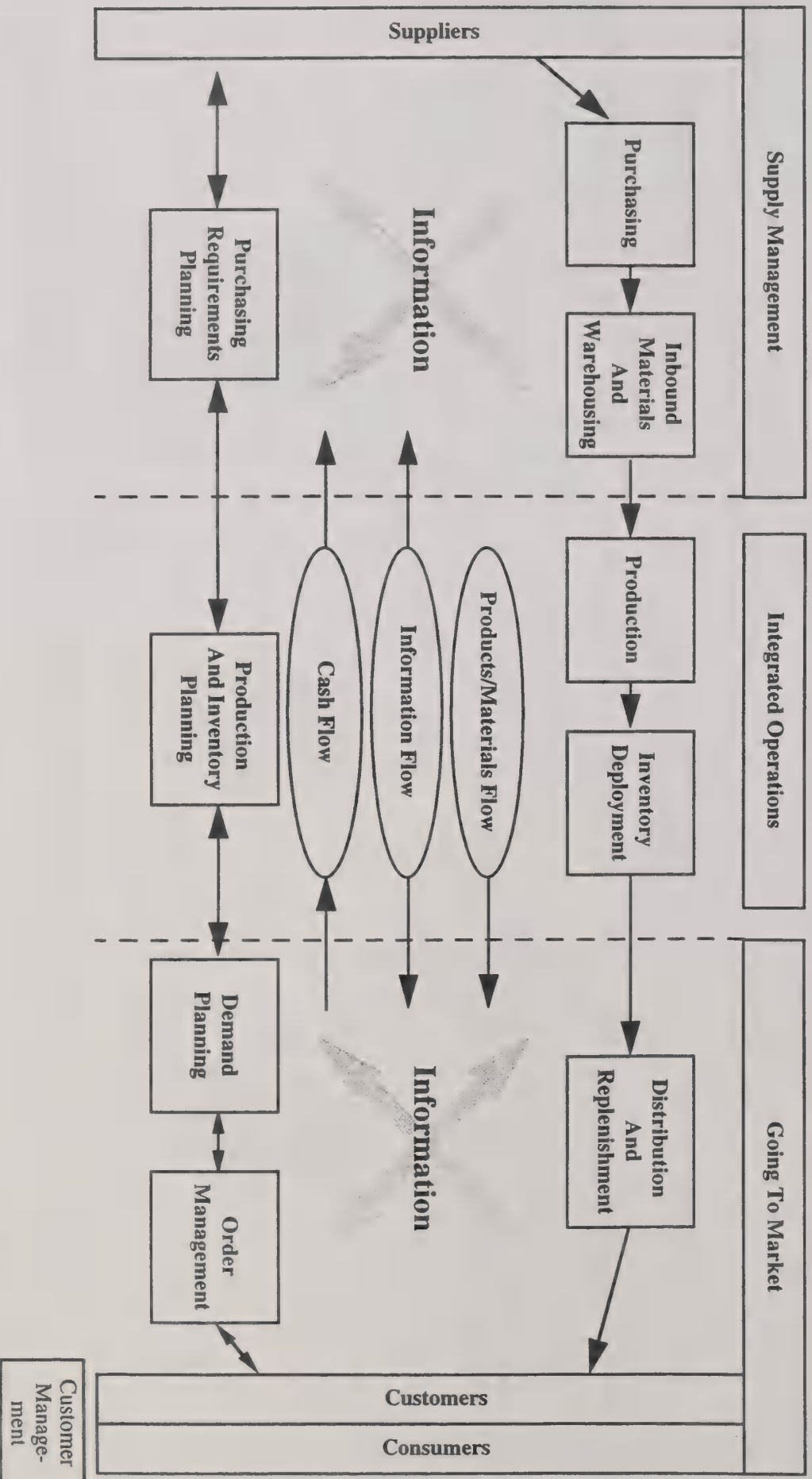


# Our approach

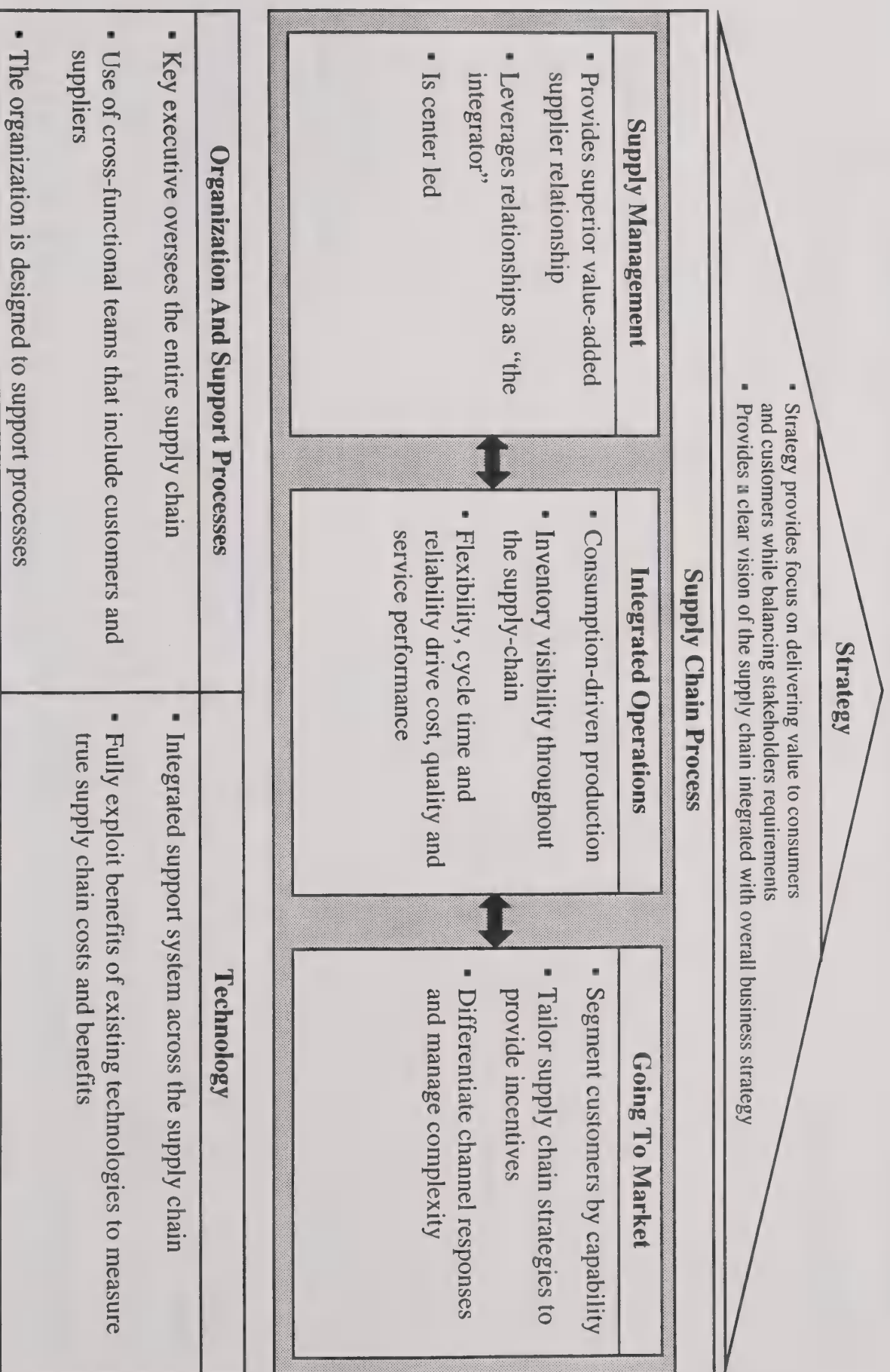


**Interactive communications between  
A.T. Kearney and client provide best results**

# Supply chain excellence is often the target for benchmarking



# Guiding principles drive supply chain integration



# Supply management through sourcing provides superior value-added supplier relationships and significant year-to-year business results

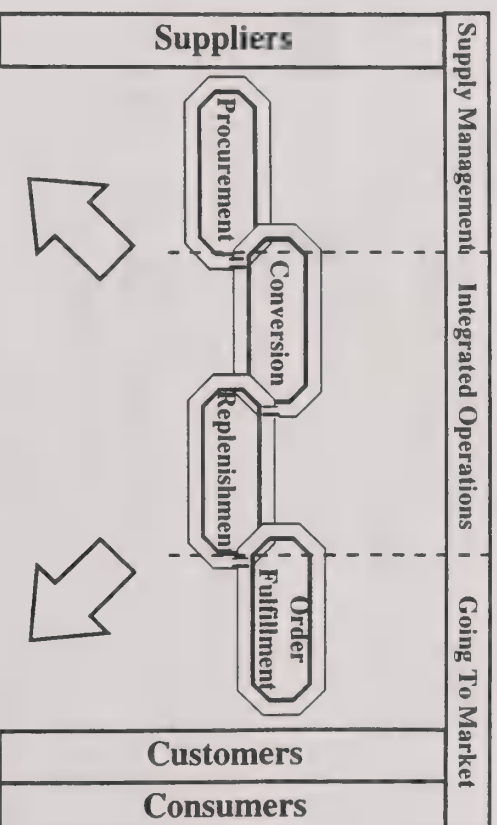


Procurement Activity	Sample Best Practices
Procurement Strategy	<ul style="list-style-type: none"> <li>• Sourcing serves as an integrator across the company</li> </ul>
Organization	<ul style="list-style-type: none"> <li>• Center-led Strategic Sourcing group evolving to an expertise center</li> <li>• Resource or accountable for all significant external relationships</li> </ul>
Sourcing Process	<ul style="list-style-type: none"> <li>• Focus on identifying leading-edge suppliers</li> <li>• Seeks a portfolio of relationships and challenges specifications and conditions during the process</li> </ul>
Supplier Management	<ul style="list-style-type: none"> <li>• Heavy resource investment in the supply base to ensure long-term development</li> <li>• Suppliers are integrated into customer's business — strategically and on a day-to-day basis</li> </ul>
Performance Measurement	<ul style="list-style-type: none"> <li>• Formalized supplier performance measurement</li> <li>• Measures address internal customer satisfaction, value creation and traditional cost, service and quality parameters</li> </ul>
Information Systems	<ul style="list-style-type: none"> <li>• Facilitate value-added analysis, speedy routine transactions and sourcing scope integration</li> </ul>

- Offset inflation plus 1% to 2% annual cost improvement
- 60%-70% of time spent on strategic activities — opportunity development, supplier selection and improvement, negotiation, etc.



# Integrated operations must streamline and align key processes consistent with demand and value requirements



## Practices

- Consumption-driven production
- Inventory visibility throughout supply chain
- Flexibility, cycle time and reliability drive cost, service and performance
- Optimum network with high-capacity utilization
- Supports speed to market/concurrent engineering
- Organized around processes and products

## Benchmarks

- On time and complete orders — 96%
- Raw material and WIP inventory — 10 days
- Finished goods inventory — 10 days
- Product changeovers — 10-20 minutes
- Manufacturing cycle time — three days
- 90% capacity utilization, based on seven days per week operations
- 94%-95% operating efficiency
- 12 to 24 months for product launch
- Three levels of organization from site manager through line workers



# “Closer-to-customer” is challenging standard practices

Pressure On ROS And ROIC

## Competitive Force

- Growth of private labels
- Maturing of categories
- New product launches by innovative competitors
- Brand extensions through packaging innovations
- Complexity of marketing
  - Database marketing
  - Category management
  - Programs tailored by account
  - New media

## Impact On “Go To Market” Strategies

- Drive to take costs out of the manufacturer/retailer interface
- A vision for growth in a mature U.S. market

## Manufacturer/Retailer Relationships

- Growth of alternative formats
  - Discount/price focus
  - Greater supply chain demand
- Consolidation of retail wholesale/industry
- Implementation of ECR initiative

## External Forces

- Changing consumer dynamics
  - Value conscious
  - Need for variety
  - Convenience conscious
- Technological advances in industry
  - Electronic commerce
  - Computer aided replenishment
  - Client/server technology

# World-class supply chain performance

Metric	Definition	Best In Class
<ul style="list-style-type: none"> <li>On-time and complete orders (%)</li> </ul>	<ul style="list-style-type: none"> <li>On time and complete = case full %<sup>N</sup> x on-time delivery %</li> </ul>	<ul style="list-style-type: none"> <li>90% to 92%</li> </ul>
<ul style="list-style-type: none"> <li>Finished-goods inventory turns (#)</li> </ul>	<ul style="list-style-type: none"> <li>Monthly cost of goods sold x 12/average inventory</li> </ul>	<ul style="list-style-type: none"> <li>10 days of supply, 25+ turns</li> </ul>
<ul style="list-style-type: none"> <li>SKUs bottom 5% of sales (%)</li> </ul>	<ul style="list-style-type: none"> <li>Percent of SKUs representing bottom 5% of total sales</li> </ul>	<ul style="list-style-type: none"> <li>15% to 30%</li> </ul>
<ul style="list-style-type: none"> <li>Cost/hundred-weight shipped (\$)</li> </ul>	<ul style="list-style-type: none"> <li>Dollars expended/100 pounds of finished products shipped</li> </ul>	<ul style="list-style-type: none"> <li>\$2.00 to \$2.50</li> </ul>

# World-class supply chain performance

Metric	Definition	Best In Class
<ul style="list-style-type: none"> <li>Defects per 100,000 produced</li> </ul>	<ul style="list-style-type: none"> <li>Number of critical and significant defects per 100,000 units produced</li> </ul>	<ul style="list-style-type: none"> <li>200 to 300</li> </ul>
<ul style="list-style-type: none"> <li>Demand adherence (%)</li> </ul>	<ul style="list-style-type: none"> <li>Proportion of time the manufacturing process can replenish market demand within a predetermined time window (seven days)</li> </ul>	<ul style="list-style-type: none"> <li>A&amp;B SKUs produced weekly, 90%</li> </ul>
<ul style="list-style-type: none"> <li>Rework (%)</li> </ul>	<ul style="list-style-type: none"> <li>Proportion of finished and in-process product requiring reconditioning due to defective materials, misformulation, etc.</li> </ul>	<ul style="list-style-type: none"> <li>0.1% to 0.5%</li> </ul>
<ul style="list-style-type: none"> <li>Weighted average manufacturing cycle time (# of days)</li> </ul>	<ul style="list-style-type: none"> <li>Average manufacturing time for A, B, C, SKUs with a 60%, 30%, 10% relative weighting</li> </ul>	<ul style="list-style-type: none"> <li>Three to five days</li> </ul>



# World-class supply chain performance

Metric	Definition	Best In Class
<ul style="list-style-type: none"> <li>Operating efficiency (%)</li> </ul>	<ul style="list-style-type: none"> <li>Effective run time/production available time</li> </ul>	<ul style="list-style-type: none"> <li>94% to 95%</li> </ul>
<ul style="list-style-type: none"> <li>Asset utilization (%)</li> </ul>	<ul style="list-style-type: none"> <li>Asset utilization = effective run time/available time</li> </ul>	<ul style="list-style-type: none"> <li>90% to 91%</li> </ul>
<ul style="list-style-type: none"> <li>Purchase-order cycle time (# of days)</li> </ul>	<ul style="list-style-type: none"> <li>Average time from placement of materials order to time of receipt by the company</li> </ul>	<ul style="list-style-type: none"> <li>Weekly releases, daily delivery, five days of supply</li> </ul>
<ul style="list-style-type: none"> <li>Average new-product development cycle time (months)</li> </ul>	<ul style="list-style-type: none"> <li>Time from initiation of concept and product development through commercial product launch</li> </ul>	<ul style="list-style-type: none"> <li>12 to 24 months</li> </ul>

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# In summary

**To Sustain And Extend Competitive Advantage, World-Class Companies Are Integrating Their Supply Chain By**



- Ensuring that the consumer is the number-one priority using tailored go-to market strategies
- Streamlining and aligning supply chain operations consistent with demand and value requirements
- Building strategic, value-added supplier and customer relationships
- Implementing comprehensive information technology strategies
- Creating measurement and reward strategies that drive improvement to the entire supply chain's true costs and benefits

---

# Our future challenge

## Rejecting

**Complacency**

“We are doing fine. Why change?”

**Resource Constraints**

“Our day-to-day business takes up all our resources.”

**Leadership**

“We are unique. Those benchmarks don’t apply.”

**Pain Of Change**

“I don’t have time to change.”





Figure 2

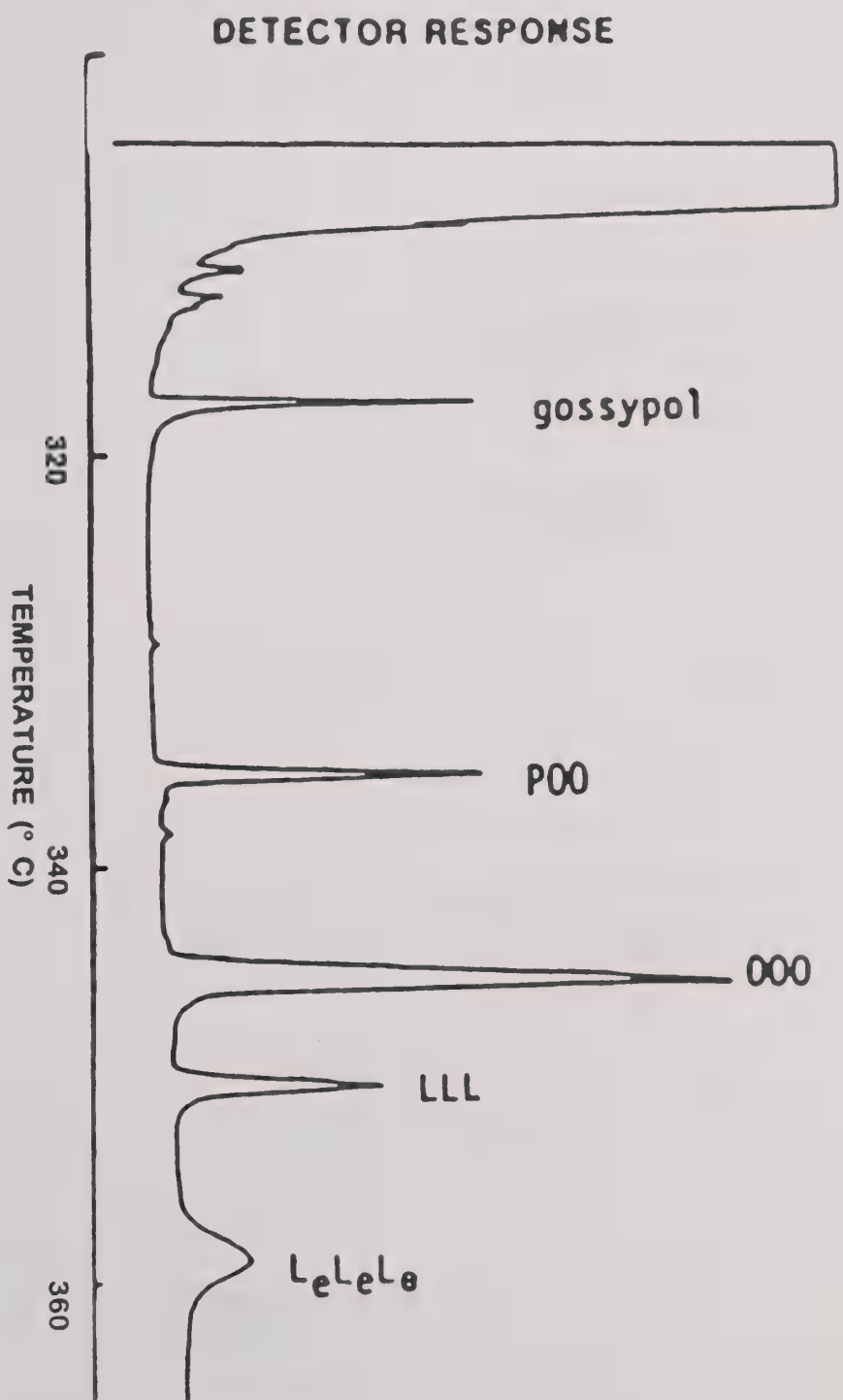


Figure 2. Capillary GC Analysis of gossypol and Oilseed TAG

Figure 3

Gas Chromatogram of SC-CO<sub>2</sub> - Extracted RBO  
(TMS derivatized RBO)  
SC-CO<sub>2</sub> @ 7000 Psi and 80°C

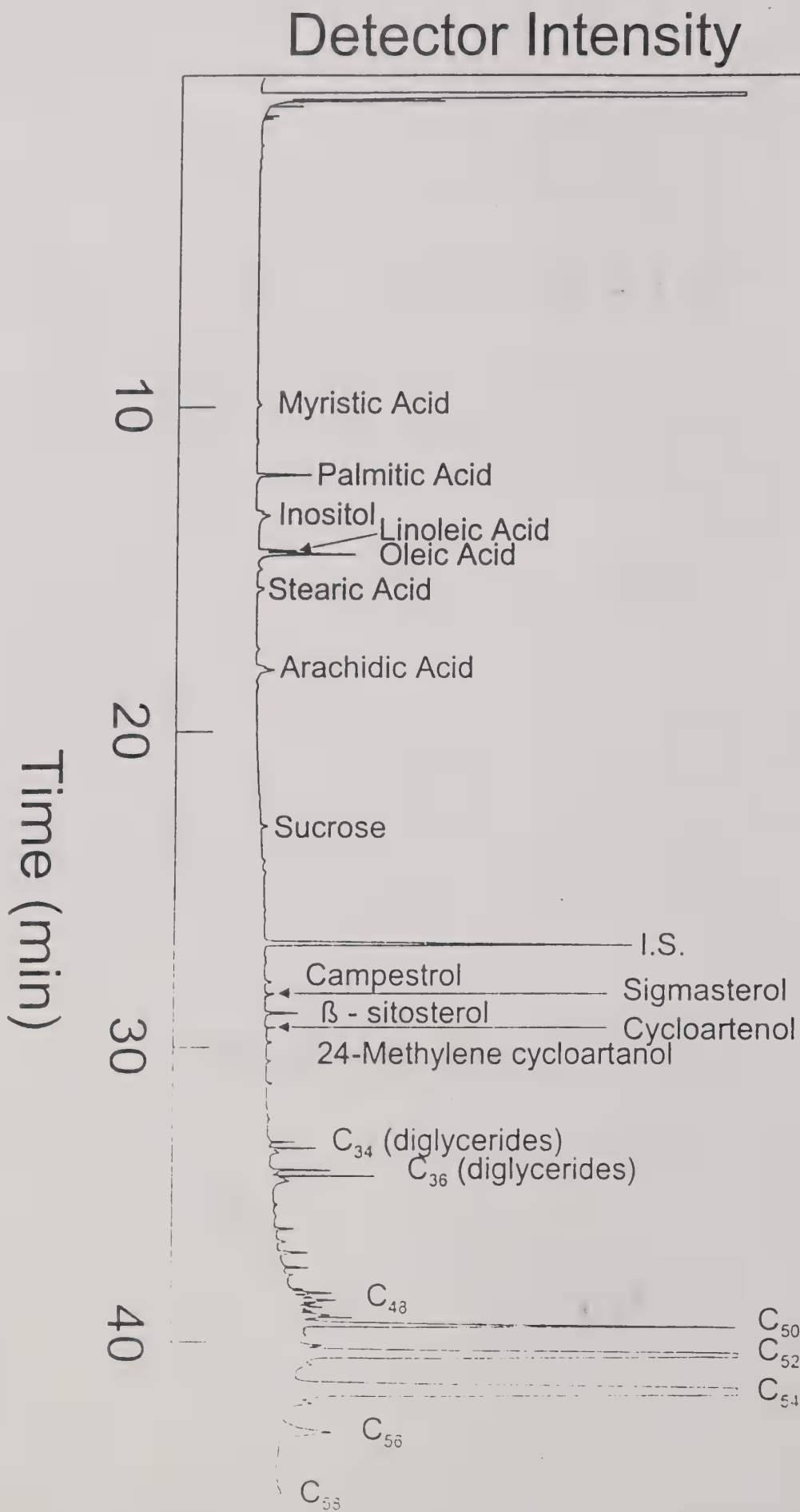
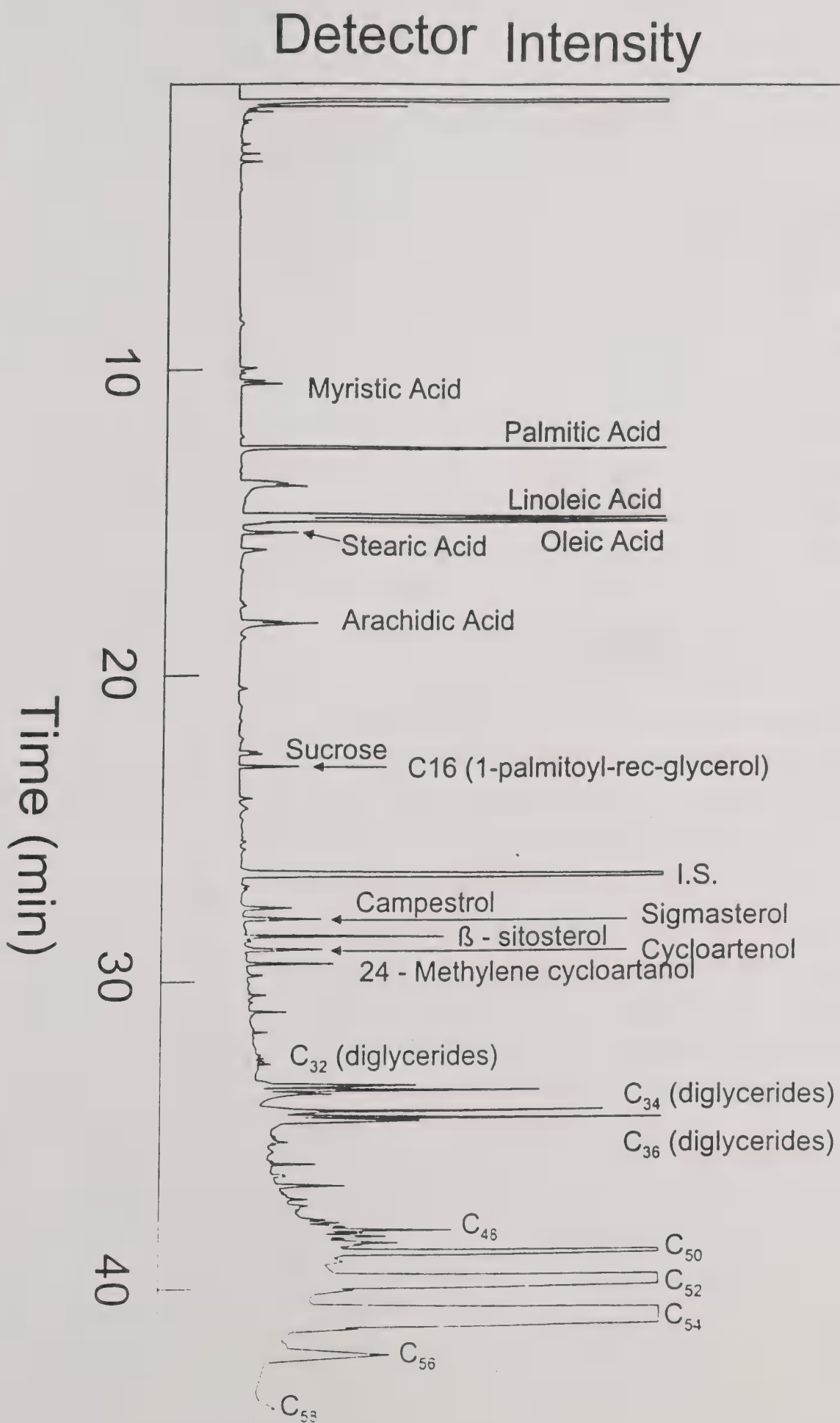


Figure 4

Gas Chromatogram of SC-CO<sub>2</sub> - Extracted RBO  
(TMS derivatized RBO)  
SC-CO<sub>2</sub> @ 9000 Psi and 100°C



# YU. V. K. 1911

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## A Recombinant Enzyme from Transgenic Soybeans: Processing and Extraction

Ann Kusnadi, Roque Evangelista and Zivko Nikolov  
Department of Agricultural and Biosystems Engineering  
Department of Food Science and Human Nutrition  
Center for Crops Utilization Research  
Iowa State University  
Ames, IA 50011

The effect of conventional soybean processing and protein extraction conditions on the recovery and activity of recombinant beta-glucuronidase (rGUS) were investigated. rGUS activity in soybeans stored at 50°C remained stable up to seven days but only for two days when stored at 70°C. The activity of rGUS was measured after each stage of conventional soybean processing. A ten percent reduction in rGUS activity was observed after the conditioning step. An additional 20% of rGUS activity was lost after hexane extraction. Processing transgenic soybeans to full-fat flour did not affect the enzyme activity. Particle size of ground soybeans did not significantly affect the extraction yield of rGUS activity. Similar amounts of rGUS were extracted with water adjusted to pH 6.0 and with a phosphate buffer of pH between 5.5 and 8.0. Greater amounts of rGUS were extracted from the full-fat than from defatted samples (flakes or flour). Protein and rGUS extraction yields were also greater at 1:30 solids-to-buffer ratio than at 1:10. rGUS was purified by an ammonium sulfate precipitation and a three-step chromatography. Native soy proteins in the extract affected the purification fold.



## A RECOMBINANT ENZYME FROM TRANGENIC SOYBEANS: PROCESSING AND EXTRACTION

Ann Kusnadi, Roque Evangelista, and Zivko Nikolov  
Department of Agricultural and Biosystems Engineering  
Department of Food Science and Human Nutrition  
Center for Crops Utilization Research  
Iowa State University, Ames, IA 50011  
and  
John Howard  
ProdiGene, College Station, TX 77845

Transgenic plants are potentially one of the most economical systems for large-scale production of proteins and peptides for industrial, pharmaceutical, veterinary, and agricultural use. Advantages of plant systems as bioreactors include low cost of growing on a large scale, easy scale-up (increase of planted acreage), natural storage organs (seeds, tubers), and established practices for efficient harvesting, transporting, storing, and processing of the plant material (Goddijn and Pen, 1995).

Several cereal and oilseed crops appear to be attractive as "factories" for recombinant protein production. The choice of crop will largely depend on the ability to efficiently recover the recombinant protein without interfering with the extraction of primary crop products (Whitelam et al., 1993). To make the transgenic plant systems commercially attractive for the production of recombinant proteins, an inexpensive way to recover and purify them needs to be established. Our long-term goal is to develop a strategy for protein recovery and purification from plant seeds that will be readily adaptable to a wide variety of proteins and at the same time allow the co-production of the traditional seed products.

Currently, transgenic soybean and corn seeds are the focus of our processing and recovery efforts. In this study, we describe the effect of conventional soybean processing and protein extraction conditions on the recovery and activity of recombinant  $\beta$ -glucuronidase (rGUS) from soybeans. The *gus* gene (codes for  $\beta$ -glucuronidase) from *Escherichia coli* was inserted into soybeans, and the rGUS produced in the seed was used in this study.

We have investigated the thermal stability of rGUS in soybeans at different temperatures. Heat stability data indicated that rGUS in the whole soybeans remained fully active for up to seven days upon incubation at 50°C. At 70°C, the enzyme activity was fully preserved for 24 h; at 90°C and 125°C no activity was measured after 12 hours and 20 min of incubation, respectively. These results suggest that seed handling and processing can be safely done at temperatures as high as 70°C as long as processing time does not exceed 24 h.

To predict the effect of processing conditions on the enzyme stability, 25 kg of transgenic seed were processed in the pilot-plant at Iowa State University by simulating conventional soybean processing for manufacturing defatted soy-flour. The major processing steps included: 1) cracking and dehulling, 2) conditioning for 20 min at 60-70°C, 3) flaking, 4) hexane extraction, 5) desolventizing room temperature and 6) milling. The activity of rGUS was analyzed after each processing stage. A ten percent reduction in rGUS activity was observed

after the conditioning step, and an additional 20% of rGUS activity was lost after the oil extraction with hexane. The milling process did not result in a significant loss of rGUS activity i.e. about 4% activity loss was measured. Transgenic soybeans were also processed to produce full-fat flour by bypassing the conditioning and oil extraction steps. The full-fat flour prepared without the conditioning and oil extraction did not show any loss of rGUS activity.

Whole soybeans were ground and fractionated into five batches with average particle size ranging between 55 and 150 mesh. No significant difference in extracted amounts of soy protein and rGUS activity was obtained. The amount of the extractable rGUS was similar when either water adjusted to pH 6.0 or sodium phosphate buffer of pH 6.0 was used. Therefore, the extraction cost could be reduced, with a minimal loss of extractable activity, by employing water instead of a buffer in the extraction of rGUS.

Solids-to-buffer ratio affected differently the soy protein and rGUS recovery. At pH 7.5, more soy protein was extracted at 1:30 solids-to-buffer ratio than at 1:10. The enzyme extraction yield was substantially affected by the solids-to-buffer ratio only when the full-fat flour and flakes were used as a starting material. For example, the amount of rGUS extracted from full-fat samples was approximately three times greater at 1:30 than at 1:10 solids-to-buffer ratio. At pH 4.5, the solid-to-buffer ratio did not play a significant role in neither rGUS nor soy protein extraction. Because soy protein amounts extracted at pH 4.5 were disproportionately reduced compared to rGUS, the enzyme specific activity in the extract was greater at pH 4.5 than at pH 7.5.

In general, more rGUS was extracted from full-fat than from defatted samples (flakes or flour) indicating that the temperature of the hexane extraction step was detrimental to rGUS integrity; defatting the soy flour with hexane at room temperature did not affect the enzyme activity.

A partial purification of rGUS from aqueous soybean extracts was achieved in three steps: ammonium sulfate precipitation, hydrophobic interaction and anion-exchange chromatography. A 150-fold purification was achieved with a 24% purification yield. The addition of an affinity chromatography step (saccharolactone resin) increased the purity by a factor of two. Further optimization of chromatography is required to match the commercial GUS preparation obtained from *E. coli* cell extracts by Sigma Chemical Co.

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# **TWO NEW METHODS FOR PRODUCING EPOXIDIZED OILS FOR INDUSTRIAL USES**

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## **Abstract**

Epoxidized vegetable oils are produced in excess of 200 million lbs per year in the US. These epoxidized oils are used mainly as stabilizers-plasticisers for PVC. Currently epoxidation is conducted on an industrial scale using hydrogen peroxide with ether acetic or formic acid (1). This procedure is not selective, and it causes corrosion problems that are related to the acidity of the generated percarboxylic acids. Additionally, the product epoxides must be separated from the acids to limit oxirane ring-opening that nevertheless causes oxirane values to be 15-20% lower than theoretical. We have investigated two new ways to introduce epoxide functionality into fats and oils. In the first method dimethyldioxirane (DMDO) is generated and used in a biphasic system with a phase transfer catalyst (2). The second method uses the enzyme lipoxygenase to generate a hydroperoxide derivative. This is rearranged by a titanium catalyst to generate an epoxy alcohol derivative. The advantage of the former procedure over currently used epoxidation methodology is that epoxidation is nearly quantitative, and removal of reactants is simplified. The advantage of the second procedure is that an epoxy alcohol is obtained that has the potential to expand industrial uses of fats and oils.

## **Introduction**

Figure 1 shows the plasticizer production from plant oils and petrochemical feedstocks in the US in 1991. Although the plant oil based plasticizers are only a fraction of total plasticizer production, they still amount to 200 million pounds per year. If better methods of preparing plant oil based plasticizer could be found, the cost of these materials would be reduced, and their market share could be increased considerably. This would allow the US to decrease its dependence on imported petroleum.

We have developed two new procedures for epoxidizing plant oils. The first procedure uses a non-acidic epoxidation reagent under phase transfer conditions (3); the second procedure uses a combination of enzymatic and chemical catalysis to produce hydroxy epoxy derivatives of oils that have the potential of expanding the usage of fats and oils as plasticisers.

## **Methods**

**Chemical catalysis of epoxide formation under phase transfer conditions (Figure 2).** A typical reaction procedure with sunflower oil is shown as follows. The oil (2.03 g, 10.7 mmol

alkene) was dissolved in 20 mL 2-butanone (methylethylketone, MEK) that was stirred vigorously with a mixture of  $\text{NaHCO}_3$  (6.56 g, 78 mmol) and 0.5 g 18-crown-6, the phase transfer catalyst (PTC). A solution of Oxone<sup>TM</sup> (Aldrich Chemical Company, Milwaukee, WI), potassium monopersulfate, (13.2 g, 21.5 mmol) in 75 mL distilled water was added over a 10 min period with the flask wrapped in foil to protect against light. After 2 h the reaction mixture was diluted with 100 mL water and extracted with 3 x 30 mL portions of diethyl ether. The combined organic extract was washed with 50 mL water and then dried over anhydrous  $\text{Na}_2\text{SO}_4$ . After removal of the solvent in vacuo, the epoxidized oil was subjected to transmethylation by treating it (0.10 g) with 0.05 g sodium methoxide in 10 mL methanol with stirring at RT for 1 h. The mixture was diluted with 20 mL water and extracted with 2 x 20 mL ether. After washing and drying over anhydrous  $\text{Na}_2\text{SO}_4$ , the ether was removed, and the product was subjected to GLC analysis.

**Epoxy hydroxy formation using sequential application of enzymatic and chemical catalysis (Figure 3).** *Methyl 13(S)-hydroperoxy-9(Z),11(E)-octadecadienoate (Me-HPODE) formation.* Linoleic acid (80 mg) was placed in a 125 mL Erlenmeyer flask along with 80 mL 0.2 M borate buffer, pH 9.0. The flask was packed in ice, and stoppered with a rubber septum. The contents were stirred gently for one-half hour while oxygen was slowly bubbled into the buffer using a metal syringe needle. Four 50  $\mu\text{L}$  aliquots of a lipoxygenase solution (32 mg/mL) in borate buffer were added at 30 min time intervals. The pH of the reaction media was lowered to 3 with 1 M HCl, and HPODE was extracted with 2 x 50 mL diethyl ether. The diethyl ether was dried over potassium sulfate, and the diethyl ether was removed under a stream of nitrogen. HPODE was dissolved in 5 mL  $\text{CH}_2\text{Cl}_2$  and treated with diazomethane to give the methyl ester.

*Titanium treatment.* Me-HPODE (27 mg, 83  $\mu\text{mol}$ ) dissolved in 4.7 mL  $\text{CH}_2\text{Cl}_2$  was treated with 63  $\mu\text{L}$  (212  $\mu\text{mol}$ )  $\text{Ti}(\text{O}-i\text{-Pr})_4$  for 1 h at 5°C. The reaction was quenched by the addition of 0.3 mL  $\text{H}_2\text{O}$ , and the reaction mixture was allowed to stand for 30 min at room temperature. The solid matter was removed by filtration through Celite. After washing the Celite with 10 mL of diethyl ether, the filtrate and wash were combined and the solvent was removed under a stream of dry nitrogen. The residue was dissolved in 6 mL  $\text{CH}_2\text{Cl}_2$ .

*Vanadium treatment.* Me-HPODE (95 mg, 291  $\mu\text{mol}$ ) dissolved in 14 mL  $\text{CH}_2\text{Cl}_2$  was treated with 26  $\mu\text{L}$  (1.5  $\mu\text{mol}$ )  $\text{VO}(\text{acac})_2$  for 1 h at 5°C. The reaction mixture was diluted with 50 mL diethyl ether and washed with water (3 x 30 mL). After drying over potassium sulfate, the solvent was removed under a stream of nitrogen.

## Results and Discussion

**Chemical catalysis of epoxide formation under phase transfer conditions.** A biphasic procedure in which a water-insoluble ketone is employed as both dioxirane reagent and organic solvent in conjunction with a phase transfer catalyst was investigated for its ability to introduce the epoxide functionality into fats and oils (3). Comparison of several ketones suggested that 2-butanone (ethylmethylketone, MEK) functioned best in this regard. Moreover, MEK is relatively insoluble in water and thus provides a phase for more intimate contact of the oxidant ethylmethyldioxirane (EMDO) with the triglycerides. In addition, the stability of EMDO with



respect to autodecomposition by radical fragmentation would be very similar to that of dimethyldioxirane and better than that of more highly substituted dioxiranes. The reactions involved in this sequence are summarized in Figure 2.

The results of an initial epoxidation of a number of fats and oils are shown in Table 1. The list of triglycerides is organized by type; namely, Group 1: low  $\alpha$ -linolenic acid residues ( $\leq 1\%$ ); Group 2: medium to high  $\alpha$ -linolenic acid content (7 to 54%) and Group 3: oils that contain less frequently encountered unsaturated fatty acids. Gas liquid chromatography was used to establish completeness of the epoxidation through loss of unsaturated fatty acids. This analysis, of course, would not permit an indication of possible polymerization due to ring cleavage. Oxirane determination (4) also is inexact because the addition of HBr to an epoxide ring can cause reaction of a neighboring epoxide ring to form a tetrahydrofuran (5). Thereby two epoxide rings are cleaved, but only a single HBr residue is consumed; ie, the stoichiometry of the standard method characteristically underestimates the oxirane content of epoxidized polyunsaturated materials. Our effort, therefore, was geared to obtaining oxirane numbers that would be better than or comparable to those of commercial samples of epoxidized soybean oil.

The low  $\alpha$ -linolenic oils and fats in Table 1 were converted to epoxidized products, and the GLC chromatograms of the corresponding methyl esters indicated little or no residual unsaturation. Corn oil, for example, was converted to an oxidized product that after transmethylation consisted of the epoxide of *cis*-9-octadecenoic acid and the diastereomeric diepoxides of *cis*, *cis*-9,12-octadecadienoic acids (5). In contrast, complete epoxidation of soya and canola oils, both of which contain significant amounts of linolenic acid (7-10%), proved difficult. Epoxidation of the soya oil with meta-chloroperbenzoic acid in methylene chloride did provide material that was free of unsaturation and that titrated near the theoretical oxirane value (89%). A commercial sample of epoxidized soybean oil likewise gave an oxirane value that was 90% of theoretical. A higher ratio of Oxone<sup>TM</sup> to oil (4:1) did not increase the epoxide content, and so we examined the epoxidations of methyl linoleate and linolenate. Using a ratio of 2.5:1 of Oxone<sup>TM</sup> to alkene (methyl linoleate) the product distribution was 29% monoepoxides, 61% diepoxides and 11% unreacted alkene; the total crude product titrated for 74% of theoretical oxirane. Rationalizing the low conversion as a slower epoxidation of the monoepoxidized material in competition with radical autodecomposition (6), the oxidant was added in two portions with an hour separating the additions. When conducted in this manner, the reaction mixture contained <2% unreacted alkene (GLC) and 83% of the theoretical oxirane content (titration). Similarly, methyl linolenate epoxidation was improved from 68% unreacted alkene to >99% triepoxides (GLC) and 87% of theoretical oxirane. Accordingly, both soybean and canola oil were epoxidized using the two-step addition of Oxone<sup>TM</sup> (total 2:1). Both epoxidized oils contained only minor amounts of residual unsaturated fatty acids and had 84% of the theoretical oxirane value. The efficiency of the two step epoxidation procedure was demonstrated further in the epoxidation of flax seed oil (54% linolenic acid). The product consists almost exclusively of the monoepoxide from oleic acid, the two diastereomeric diepoxides from linoleic acid and the four diastereomeric epoxides from linolenic acid and the unreactive saturated acids, palmitic and stearic. An example of the complete epoxidation of other oils in Table 1 is that of castor oil, where the ricinoleic acid was oxidized to the diastereomeric epoxides of (R)-12-hydroxy-*cis*-9-octadecenoic acid. As noted previously, biphasic oxidation favors epoxidation of the double

bond over oxidation of the alcohol to the ketone (7). On the other hand, biphasic oxidation of tung oil, whose major fatty acyl component is  $\alpha$ -eleaostearic acid a conjugated polyunsaturated fatty acid, gave a product with the lowest oxirane value. Furthermore, GLC showed little unreacted unsaturated acyl groups and only small amounts of epoxide products indicating that tung oil triglycerides were polymerized rather than epoxidized.

Additional reaction parameters were evaluated to determine their importance to the epoxidation rate and the degree of conversion of the oils. Not surprisingly, the heterogeneous reaction mixture responded to those parameters normally associated with interfacial contact. Although an increase in the PTC, 18-crown-6, increased the rate of reaction, use of a Florence flask with vigorous magnetic stirring produced the same high conversions in about 1 h without additional PTC. The epoxidation of methyl oleate was conducted with varying amounts of Oxone<sup>TM</sup> under otherwise standardized conditions (nature of reaction vessel, agitation, amounts of solvents, PTC, NaHCO<sub>3</sub>, and rate of addition of the oxidant) and conversion to epoxidized ester was monitored by GLC. Approximately 75% of the oxidizing power of the Oxone<sup>TM</sup> was used during the first 30 min of reaction time counting the 10 min interval employed for the addition. Reactions would be essentially complete, therefore, in 1-2 h. This also indicated that more effective use could be made of the oxidant were it added in portions rather than at one time as noted above. Also, the amount of NaHCO<sub>3</sub> required for effective reaction depends upon maintaining a sufficiently high pH to sustain nucleophilic oxygen (persulfate anion). Reactions employing decreasing amounts of bicarbonate indicated consistent success with a 2.5:1 molar ratio of NaHCO<sub>3</sub> to Oxone<sup>TM</sup>.

Other PTC's that served well included tetrabutylammonium chloride and bisulfate, and Aliquat<sup>TM</sup> 336. These were added in the same weight amounts as was the 18-crown-6 in the epoxidation of olive oil. Results from these reactions were not significantly different from those given in Table 1. The use of ammonium salts in place of crown ethers has notable advantages, since the salts are generally less expensive, are less toxic, and are recoverable from the reactions. Biphasic reactions of dioxiranes were conducted using organic solvents other than the ketone from which the dioxirane was prepared. In Table 2 is shown the degree of conversion achieved for the oxidation of soybean oil with several cosolvents. In each case the 2-butanone was present in 10-fold molar excess to the ester. In none of these cases was the conversion greater than when 2-butanone was employed as the solvent.

In summary, we studied the epoxidation of unsaturated fats and oils using Curci's biphasic method (3) employing 2-butanone as solvent and ethylmethyldioxirane as oxidant. The best conversions of polyunsaturate-containing oils were obtained with two-step addition of oxidant (Oxone<sup>TM</sup>) with a molar ratio of oxidant to oil of 2.5:1. Moreover, epoxidation reactions also were successful when quaternary ammonium salts were used as phase transfer catalyst.

**Epoxy hydroxy formation using sequential application of enzymatic and chemical catalysis.** Me-HPODE was incubated with Ti(O-*i*-Pr)<sub>4</sub> for 1 h at 5°C. Analysis of the products by normal phase HPLC revealed a simple profile (Figure. 4). The peaks corresponding to minor products A were collected together. Their mass spectra were consistent with these being a mixture of methyl linoleate and conjugated methyl linoleate.

Product B comprised approximately 30% of the total product. Product B was identified as



methyl 13-hydroxy-9,11-octadecadienoate by comparison of its mass spectra to that of an authentic standard purchased from Oxford Biomedical Research (Oxford, MI).

Major product C accounted for approximately 67% of the total product. Its mass spectrum showed an ion at  $m/z$  288 (M-[18+31]). After formation of the  $(\text{CH}_3)_3\text{Si}$  derivative, its mass spectrum showed ions at  $m/z$  383 (M-15), 367 (M-31), 327 (M-71), 270 (M-128; rearrangement with expulsion of  $\cdot\text{CO}-\text{CH}(\text{O}\cdot)-\text{CH}_2)_4-\text{CH}_3$ ) (8) and 173 ( $(\text{CH}_3)_3\text{SiO}^+=\text{CH}-(\text{CH}_2)_4-\text{CH}_3$ ). The UV/VIS spectrum showed end absorbance below 210 nm. Thus the data show that compound C is a monounsaturated 18 carbon epoxy alcohol methyl ester containing the hydroxyl group at C-13.

The neat infrared spectrum of product C showed a broad band centered at  $3425\text{ cm}^{-1}$  (hydrogen bonded hydroxyl),  $1739\text{ cm}^{-1}$  (ester carbonyl), and  $890\text{ cm}^{-1}$  (*trans* epoxide). No absorption band appeared in the region  $900\text{--}1000\text{ cm}^{-1}$ , excluding the presence of *trans* double bond(s) (9).

The decoupled  $^{13}\text{C}$  NMR (100 MHz) spectrum of product C obtained in  $\text{C}_6\text{D}_6$  showed important signals at  $\delta$  51.6 ( $\text{OCH}_3$ ), 52.9 (C-12), 63.7 (C-11), 71.6 (C-13), and 174.2 ( $\text{C}(\text{O})\text{OCH}_3$ ). The solvent signal partially obscured those from the double bond carbons. Thus the  $^{13}\text{C}$  NMR spectrum was also obtained in  $\text{C}_4\text{D}_8\text{O}_2$  to give the signals for the double bond:  $\delta$  129.4 (C-10) and 137.5 (C-9). Since there are only two signals for the epoxide carbons, two signals for the double bond carbons, and one signal for the alcoholic carbon, product E is predominantly one structural isomer.

The  $^1\text{H}$  NMR ( $\text{C}_6\text{D}_6$ , 400 MHz) spectrum of product C showed important signals at  $\delta$  2.97 (dd,  $J = 2.2, 4.6\text{ Hz}$ , 1H, H-12), 3.60 (br s, 1H, H-13), 3.88 (dd,  $J = 2.3, 8.7\text{ Hz}$ , 1H, H-11), 5.39 (dd,  $J = 9.0, 11.0\text{ Hz}$ , 1H, H-10), 5.80 (dt,  $J = 7.5, 11.2\text{ Hz}$ , 1H, H-9). The coupling constant  $J_{9,10}$  was 11–11.2 Hz, demonstrating that the double bond is in the *cis* configuration:  $J = 5\text{--}14\text{ Hz}$  for *cis* protons and 12–18 Hz for *trans* protons (9). The coupling constant  $J_{11,12}$  was 2.2–2.3 Hz, demonstrating that the configuration of the epoxide group is *trans*:  $J = 4.3$  for *cis* and 2.1–2.4 for *trans* (10). The coupling constant  $J_{12,13}$  is 4.6 Hz, indicating that the relationship between the adjacent protons of the alcohol and the epoxide is *threo*:  $J = 5$  for *threo* and 3.25 for *erythro* (11). An analogous coupling constant reported for the *threo* derivative of an alcoholic epoxide derived from the action of the fungus, *Saprolegnia parasitica*, upon arachidonic acid was 4.5 Hz (12). In support of the *threo* assignment H-13 resonates at 3.60 ppm:  $3.8 \pm 1\text{ ppm}$  for *erythro* and  $3.5 \pm 1\text{ ppm}$  for *threo* (13).

From all of the data it is concluded that the stereochemical structure of product C is methyl 11(*R*),12(*R*)-epoxy-13(*S*)-hydroxy-9(*Z*)-octadecenoate (Figure 5).

Minor product D is formed from C when the epoxide is hydrolyzed. This was inadvertently shown when the reaction was conducted with a wet sample of Me-HPODE: Product D was the major product formed. The mass spectrum of the  $(\text{CH}_3)_3\text{Si}$  derivative of D and its UV/VIS spectrum demonstrated that product D is a 18 carbon trihydroxy methyl ester with a single double bond.

Larger amounts of product were subjected to HPLC analysis, and minor peaks were collected in order to determine if the *erythro* or other structural epoxy alcohol isomers were being formed. No other isomers were detected. One minor product identified was methyl 13-keto-9,11-octadecadienoate (14).

As an additional check on the findings, Me-HPODE was subjected to the action of  $\text{VO}(\text{acac})_2$ ,

since prior work had shown that both the *erythro* and *threo* epoxy alcohol isomers were formed by the action of this catalyst (15). HPLC and GC-MS analysis of the products derived from the action of VO(acac)<sub>2</sub> showed that two products had mass spectra identical to that of epoxy alcohol C. The *threo* epoxy alcohol eluted at 17.6 min, and the *erythro* isomer (vide ante) eluted at 14.6 min. The *erythro* and *threo* isomers comprised approximately 70% of the product, and the ratio of *erythro* to *threo* was 1.0:1.1. A minor product had a mass spectrum identical to that of alcohol B.

The results of the study with VO(acac)<sub>2</sub> showed that the *erythro* isomer of the epoxy alcohol and the alcohol B elute very closely during HPLC analysis. If the *erythro* isomer of the epoxy alcohol had been produced by Ti(O-*i*-Pr)<sub>4</sub> catalysis, then this isomer would have been collected along with alcohol B when sample was obtained for NMR analysis. Examination of the <sup>13</sup>C NMR of alcohol B in the region where epoxide carbons resonate revealed no discernible signals. From consideration of the signal to noise ratio, an estimate for the upper limit of the yield of the *erythro* isomer by Ti(O-*i*-Pr)<sub>4</sub> is 3%. Thus the results demonstrate that Ti(O-*i*-Pr)<sub>4</sub> is more *threo* selective than VO(acac)<sub>2</sub>. There have been few studies on the direct action of Ti(O-*i*-Pr)<sub>4</sub> on unsaturated hydroperoxides. However, the transfer of oxygen from saturated hydroperoxide to α,β-unsaturated alcohol has been intensively studied (16). These studies also showed that Ti(O-*i*-Pr)<sub>4</sub> is more *threo* selective than VO(acac)<sub>2</sub> (17).

Recent work has focused on the commercial development of plant species that contain unique fatty acid compositions (18). Two such species are *Lesquerella* and *Vernonia* which produce lesquerolic acid (14-hydroxy-*cis*-11-eicosenoic acid) and vernolic acid (12,13-epoxy-*cis*-9-octadecenoic acid), respectively. The alcohol epoxy material produced by the Ti(O-*i*-Pr)<sub>4</sub> rearrangement of Me-HPODE can be considered to be a hybrid of these two acids, and it has valuable properties that will make it uniquely useful for industrial use.

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**TABLE 1**  
**Epoxidation of Fats and Oils with Ethylmethyldioxirane<sup>a</sup>**

	Conversion <sup>b</sup>	Oxirane % <sup>c</sup>	Unsaturated Fatty Acyl Content(%)		
			Mono <sup>d</sup>	Di <sup>e</sup>	Tri <sup>f</sup>
<u>Group 1</u>					
Coconut	100	92	6	2	trace
Tallow	100	90	44	3	1
Corn	100	84	25	61	1
Olive	100	96	79	8	1
Safflower	100	88	13	78	trace
Sunflower	100	84	20	69	0
<u>Group 2</u>					
Soya	73	62	24	54	7
	99 <sup>g</sup>	89			
Canola	75	68	58	25	10
	99 <sup>g</sup>	90			
Rapeseed	96 <sup>g</sup>	88	60 <sup>h</sup>	12	7
Flaxseed	78	71	21	16	54
	99 <sup>g</sup>	92			

Group 3	Conversion <sup>b</sup>	Oxirane % <sup>c</sup>	Unsaturated Fatty Acyl Content(%)		
			Mono <sup>d</sup>	Di <sup>e</sup>	Tri <sup>f</sup>
Castor	100	97	94 <sup>i</sup>	4	0
Coriander	100	81	80 <sup>j</sup>	15	trace
Meadowfoam	100	91	71, <sup>k</sup>	20 <sup>l</sup>	0
Tung	97	38	7	5	82 <sup>m</sup>

<sup>a</sup>These reactions used 2.0:1 Oxone<sup>TM</sup> to alkene equivalents.

<sup>b</sup>% Conversion of unsaturated fatty acids in the starting fat or oil to epoxides as determined by GLC of methyl esters.

<sup>c</sup>Determined by HBr/acetic acid titration (7).

<sup>d</sup>Oleic acid.

<sup>e</sup>Linoleic acid.

<sup>f</sup> $\alpha$ -Linolenic acid.

<sup>g</sup>Two-step addition of Oxone<sup>TM</sup>.

<sup>h</sup>cis-13-Docosenoic acid (47%).

<sup>i</sup>12-Hydroxy-cis-9-octadecenoic acid (87%).

<sup>j</sup>cis-6-Octadecenoic acid (70%).

<sup>k</sup>cis-5-Eicosenoic acid (61%) + cis-13-docosenoic acid (10%).

<sup>l</sup>cis, cis-5,13-Docosadienoic acid (20%).

<sup>m</sup>cis, trans, trans-9,11,13-Octadecatrienoic acid (82%).

TABLE 2

**Phase Transfer Catalyzed Epoxidations of Soy Oil with Co-solvents<sup>a</sup>**

Solvent	Conversion % <sup>b</sup>
2-butanone	73
chloroform	35
1,2-dichloroethane	44
methylene chloride	65

<sup>a</sup>Reactions were conducted with one-step addition of Oxone<sup>TM</sup> (see Materials and Methods) substituting the indicated solvent for 2-butanone and using 10 equivalents of the ketone to generate the dioxirane.

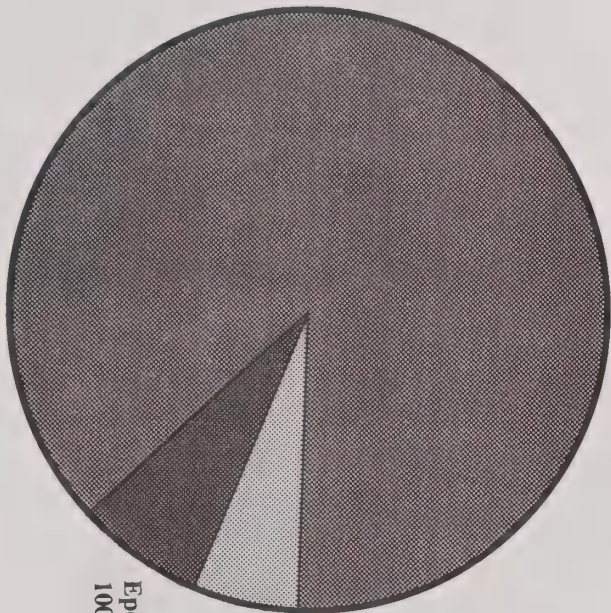
<sup>b</sup>Determined by GLC of methyl esters.

### Figure Legends

- Figure 1. Plasticizer production from plant oils and petrochemical feedstocks in the US.
- Figure 2. Generation of ethylmethyldioxirane (EMDO) and its use in methylethylketone (MEK) to convert unsaturated fats to epoxy derivatives.
- Figure. 3. Generation of Methyl 13(*S*)-hydroperoxy-9(*Z*),11(*E*)-octadecadienoate (Me-HPODE) from oxygen using the enzyme lipoxygenase (LOX), and its conversion to alcohol epoxide using  $\text{Ti}(\text{O-}i\text{-Pr})_4$ .
- Figure 4. Normal phase HPLC analysis of the products obtained by the treatment of methyl 13(*S*)-hydroperoxy-9(*Z*),11(*E*)-octadecadienoate with  $\text{Ti}(\text{O-}i\text{-Pr})_4$ . Structural analyses, as discussed in the text, resulted in the following elucidations: A, methyl linoleate and its conjugated isomer; B, methyl 13(*S*)-hydroxy-9(*Z*),11(*E*)-octadecadienoate; C, methyl 11(*R*),12(*R*)-epoxy-13(*S*)-hydroxy-9(*Z*)-octadecenoate; D, trihydroxy hydrolysis product of C.
- Figure 5. Absolute configuration of alcohol epoxide arising from the rearrangement of Me-HPODE with  $\text{Ti}(\text{O-}i\text{-Pr})_4$ .



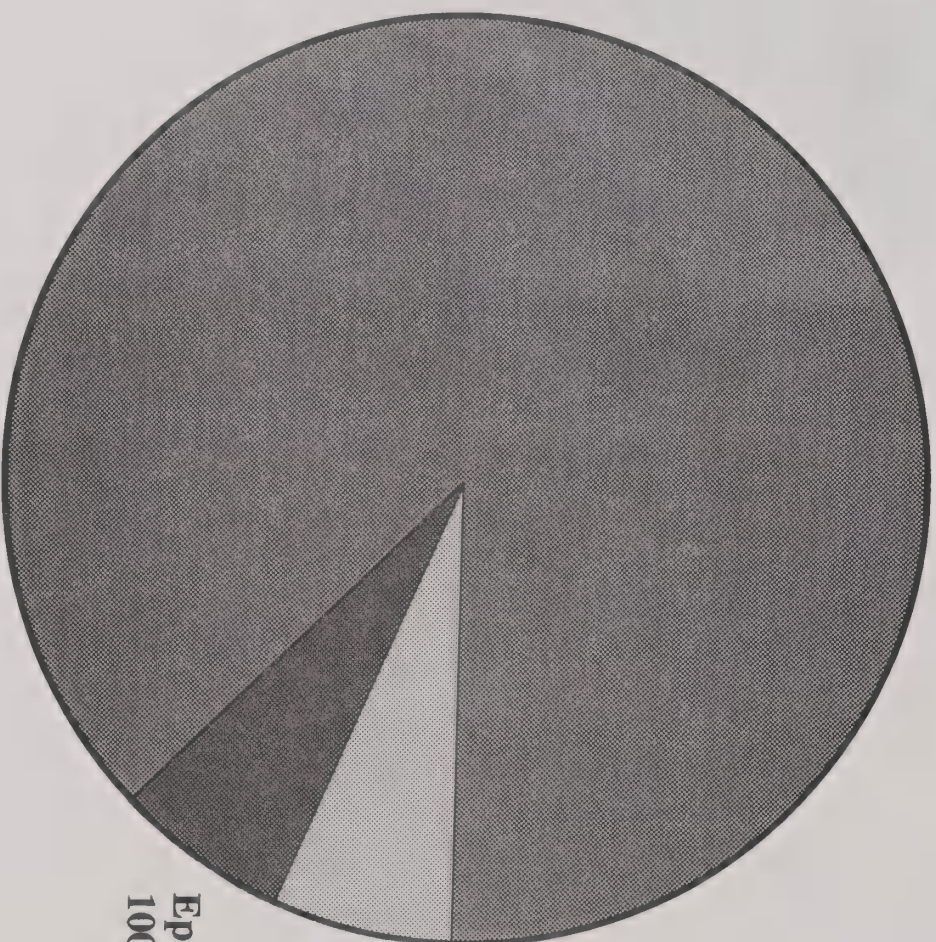
Petrochemical Plasticizers  
1324 MM lb.



Other Plant-Based  
Plasticizers, 100 MM lb.

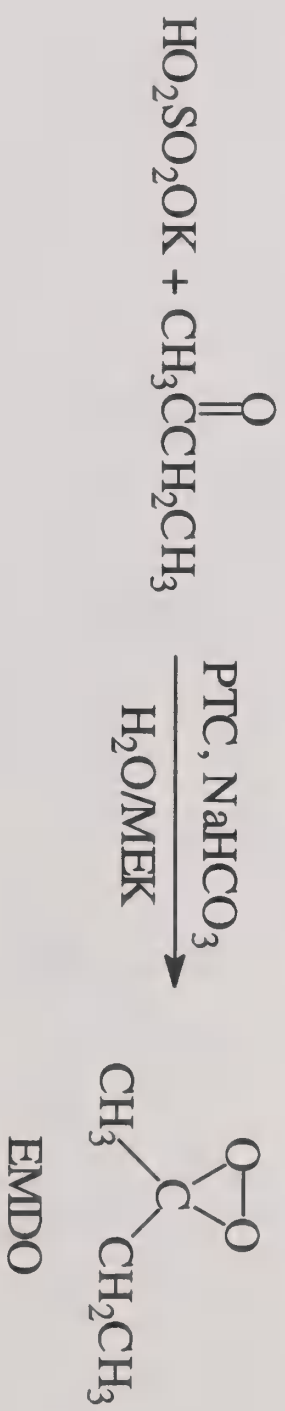
Epoxidized Soybean Oil  
100 MM lb.

**Petrochemical Plasticizers**  
**1324 MM lb.**



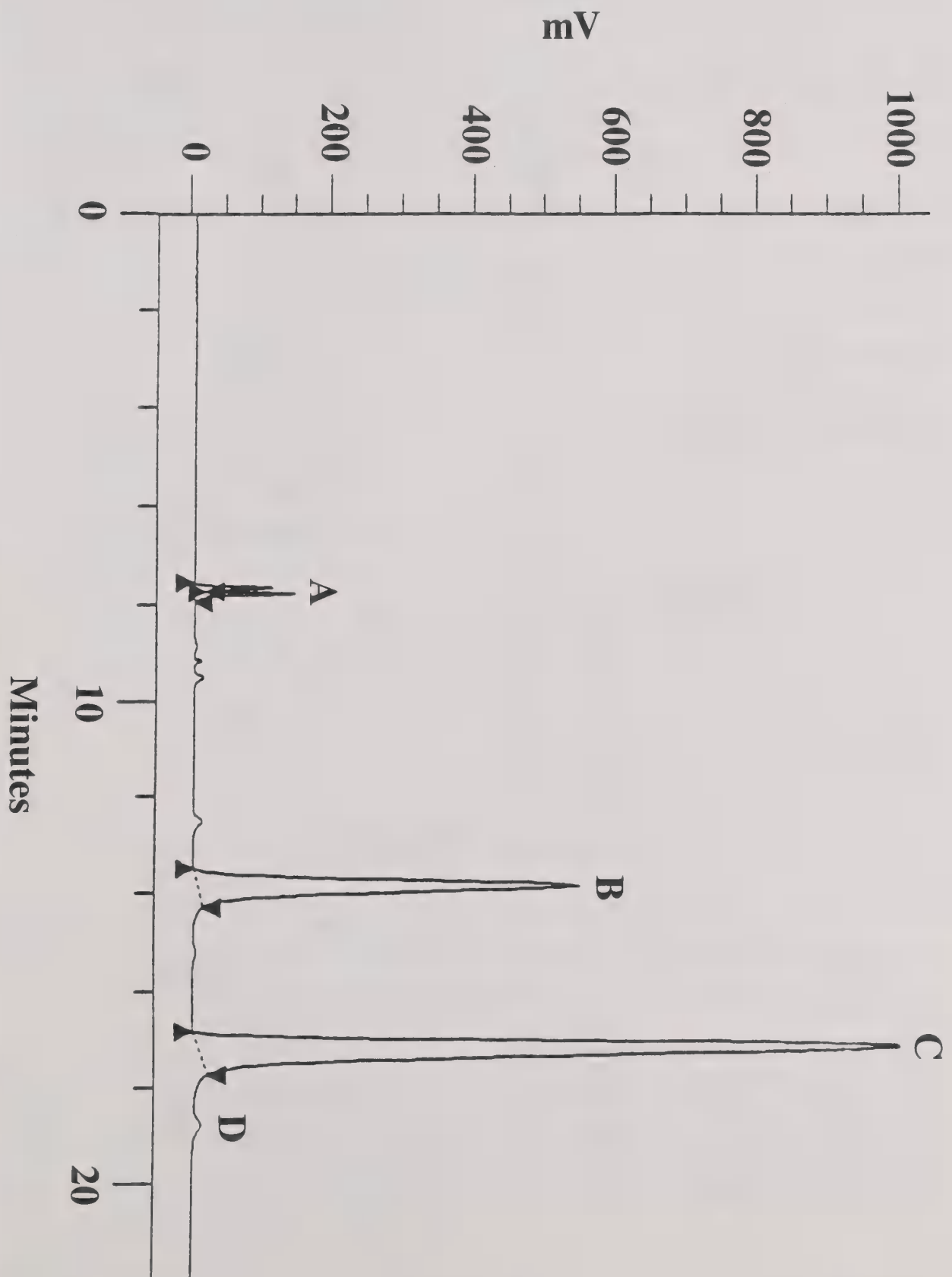
**Other Plant-Based  
Plasticizers, 100 MM lb.**

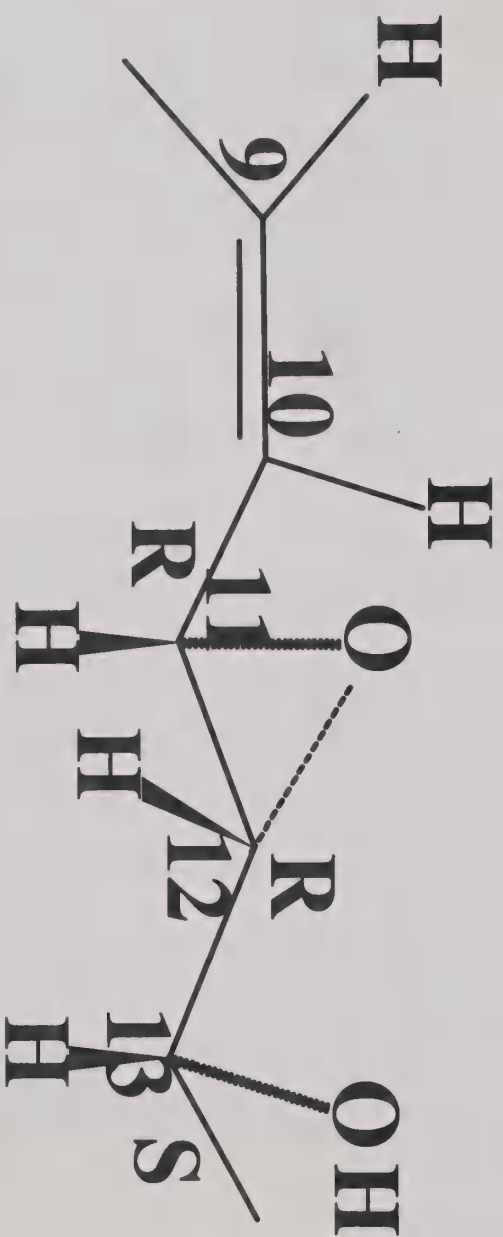
**Epoxidized Soybean Oil**  
**100 MM lb.**











Proton	Chemical Shift	Appearance of Spectrum	Coupling Constants
H-9	5.80 ppm	dt	7.5, 11.2 Hz
H-10	5.39 ppm	dd	9.0, 11.0 Hz
H-11	3.88 ppm	dd	2.3, 8.7 Hz
H-12	2.97 ppm	dd	2.2, 4.6 Hz
H-13	3.60 ppm	br s	

# Soapstock composition and its impact on the quality of cottonseed meal

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Except for a few small plants that use expellers, most cottonseed oil mills in the United States have converted to expanders to prepare kernels for oil extraction. These mills also incorporate miscella refining to increase productivity and to improve oil quality.

Soapstock is the main co-product of the refining process. Because of the limited markets for this material, most soapstock is blended with the defatted meal (marc) prior to solvent recovery and toasting.

If high quality seed is milled, the quantity of soapstock produced during refining is relatively small and the effect of added it to the extracted marc is minimal and predictable. Seed quality, though, is affected by weather condition before and during harvest. Wet weather activates lipases, which hydrolyze triglycerides to form free fatty acids (FFAs). The FFA content of high quality seed is typically less than 1%. In contrast, the FFA content of low quality seed can be above 10%. In addition to the degradation of oil that results because of lipase activity, the high FFAs increase the production of soapstock and the loss of neutral oil in soapstock. Addition of this added soapstock can reduce the percentage of meal protein below commercial specifications, which can only be compensated for by a reduction in the percentage of added hulls or high protein additives.

The composition of soapstock generated from several oil mills during the 1994-1995 processing season was studied. Soapstock from these mills averaged 41-45% moisture and residual solvent. On a dry basis, the material contained 47-52% sodium soap, 10-37% neutral oil, 4.2-10.5% gossypol, and 11.3-14.3% ash.

To ensure the composition of cottonseed meal, mills should account for the meal variation associated with the addition of soapstock. A discussion of the effects of adding soapstock to meal is presented, and general guidelines are given to regulate meal quality during times when poor quality seed must be processed.



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Pulliam, Michael	C & T Quincy Foods	Quincy, IL, USA
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Simone, Curtis	Yazoo Valley Oil Mill Inc	Greenwood, MS, USA



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Oliver, Don	Buckeye Cellulose Corp	Memphis, TN, USA
Farmer, Richard	Bunge Corp	St Louis, MO, USA
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Van Hulle, John	Caschem Inc	Bayonne, NJ, USA
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Wedegaertner, Tom	Cotton Inc	Raleigh, NC, USA
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Smith, Philip	Crown Iron Works Co	Minneapolis, MN, USA
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Leclef, Etienne	De Smet Process & Technology	Atlanta, GA, USA
Middleton, Scott	Delta Oil Mill	Jonestown, MS, USA
Kerr, Phil	Du Pont Optimum Quality Grains	Des Moines, IA, USA
Kemper, Tim	French Oil Mill Machinery Co	Piqua, OH, USA
Stroup, Bob	French Oil Mill Machinery Co	Piqua, OH, USA
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Jones, Lynn	Natl Cottonseed Products Assn	Memphis, TN, USA
Kinard, David	Natl Cottonseed Products Assn	Memphis, TN, USA
Bell, Maurice	Noctorum Inc	London, ON, Canada
Wells, Mike	Nutrition Technology Corp	Abbeville, LA, USA
Ball, David	Oil-Dri Corp of America	Chicago, IL, USA
Cenac, Greg	Oil-Dri Corp of America	Chicago, IL, USA
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Farr, Walter	Owensboro Grain Co	Owensboro, KY, USA
Jewell, Harold	Owensboro Grain Co	Owensboro, KY, USA
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Detamore, Lance	Producers Coop Oil Mill	Oklahoma City, OK, USA
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Rhee, K C	Texas A & M University	College Station, TX, USA
Williams, Gary	Texas A & M University	College Station, TX, USA
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